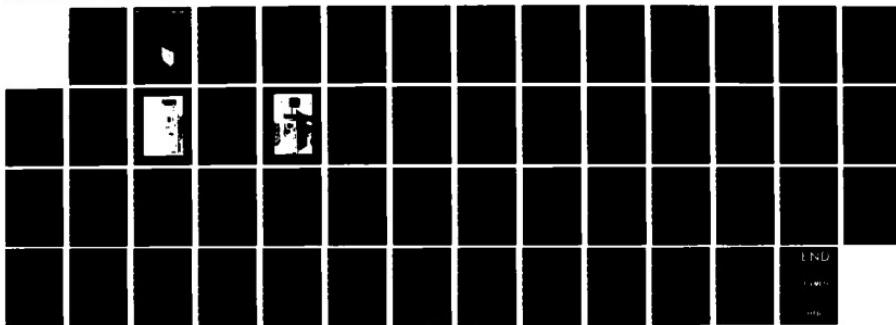


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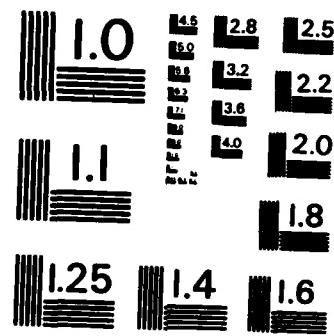
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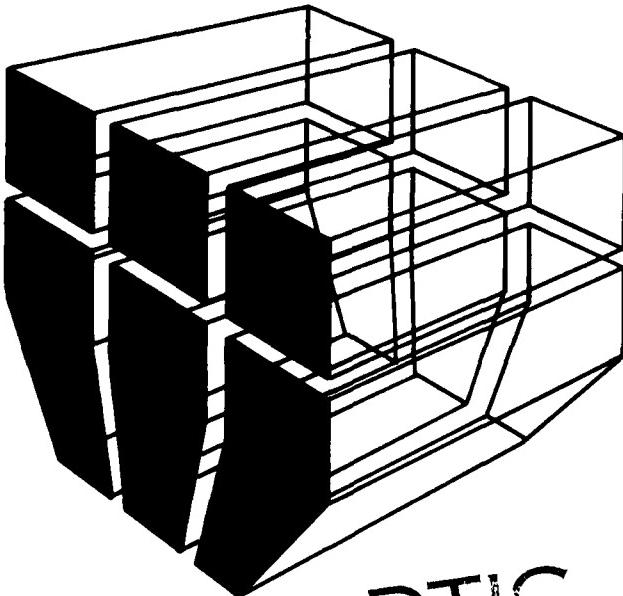
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July 1985  
Advanced Concepts for Quality Assurance

AD-A159 148

# Operations Guide and Modification Analysis for Use of the CE Concrete Quality Monitor on Roller-Compacted Concrete and Soil Cement

by  
Debbie J. Lawrence

The Corps of Engineers Concrete Quality Monitor (CE CQM) has been modified to determine water and cement contents of roller compacted concrete (RCC) and cement content of soil cement. The CE CQM makes it possible to judge the quality of RCC and soil cement as it is being placed, thereby helping avoid the high cost of replacing defective material.



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## **FOREWORD**

This work was performed for the Directorate of Engineering and Construction, Office of the Chief of Engineers (OCE), under Project 4A162731AT41, "Military Facilities Engineering Technology"; Task Area B, "Construction Management and Technology"; Work Unit 029, "Advanced Concepts for Quality Assurance." The OCE Technical Monitor was Donald Samanic, DAEN-ECC-C.

The research was performed by the Engineering and Materials Division (EM), U.S. Army Construction Engineering Research Laboratory (USA-CERL). Dr. R. Quattrone is Chief of USA-CERL-EM.

Appreciation is extended to Walla Walla District for cooperation in collecting and testing the data for CQM use with roller-compacted concrete (especially Dennis Baird, Laboratory Chief, and William Street, both at the Willow Creek Laboratory, and E. Schrader, dam designer). Appreciation is also extended to the Tulsa District for collecting data for CQM use with soil cement (especially to B. Collins, Dennis Duke, and Truscott Laboratory personnel).

COL Paul J. Theuer is Commander and Director of USA-CERL, and Dr. L. R. Shaffer is Technical Director.

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## **OPERATIONS GUIDE AND MODIFICATION ANALYSIS FOR USE OF THE CE CONCRETE QUALITY MONITOR ON ROLLER-COMPACTED CONCRETE AND SOIL CEMENT**

### **1 INTRODUCTION**

#### **Background**

The U.S. Army Corps of Engineers (CE) Tulsa District has been using an American Association of State Highway and Transportation Officials (AASHTO) test method<sup>1</sup> to determine the cement content of soil cement for construction. This method, although usually reliable, did not provide acceptable results at a Truscott, TX, project.

Another Corps District, Walla Walla, had identified a need for a method to determine the water and cement contents of roller-compacted concrete (RCC). During Walla Walla's construction of the Corps' first all-RCC dam (Willow Creek), it was found that a nuclear density meter could be used to track water, but that no method existed to rapidly determine cement content onsite. One solution was to send minus-four samples (RCC sieved through a No. 4 sieve) to Portland District's laboratory for evaluation. However, this involved too much time and tested only minus-four samples—not the whole mixture.

The Tulsa and Walla Walla Districts were both interested in testing the CE's Concrete Quality Monitor (CE CQM) for use with soil cement and RCC, respectively. The CE CQM water test consists of mixing a known weight of concrete with a salt solution of known volume and strength. The strength of the intermixed salt solution is then determined and directly related to the water in the concrete sample. The CE CQM cement test consists of separating the aggregate from the cement, uniformly suspending the cement in dilute nitric acid, and determining the calcium strength of the dissolved solution. This calcium content is proportional to the concrete's cement content. Both tests have proven to be rapid (the water test takes 3 to 4 min and the cement test takes 6 to 7 min), simple, field-worthy, and reliable. Appendix A gives a detailed description of the equipment and procedures.

<sup>1</sup> American Association of State Highway and Transportation Officials (AASHTO) Method T 144-74, *Standard Method of Test for Cement Content of Soil-Cement Mixture* (1974).

The CE CQM is the third generation of a method originally proposed by R. T. Kelly and J. W. Vail of the Greater London Council.<sup>2</sup> All three generations use the water and cement tests described above, but vary the equipment and analytical technique. The original Kelly-Vail (KV) method (Generation 1) used volumetric chloride ion titration to determine the water content and flame photometry (calcium signature) to determine cement content.<sup>3</sup> The USA-CERL/KV method (Generation 2) did the calcium analysis (cement test) by titrating with an ethylene diamine tetraacetate (EDTA) solution in the presence of a buffer and an eriochrome black-T indicator.<sup>4</sup> The CE CQM system (Generation 3) uses slightly different equipment to separate the aggregate and cement to obtain a representative sample of the cement suspension. It includes a commercially available calcium analyzer and chloride meter to determine calcium and chloride solution strengths, respectively.<sup>5</sup> Because of its experience with the CE CQM, the U.S. Army Construction Engineering Research Laboratory was asked to test this method for use with soil cement and RCC.

#### **Purpose**

The purpose of this report is to describe (1) the steps necessary to determine if the CE CQM can accurately and reliably test the required job materials, (2) the modifications needed for CE CQM operation with soil cement and roller compacted concrete, and (3) results of the laboratory and field tests on the soil cement and RCC using the modifications.

#### **Approach**

Samples of soil and cement from the Truscott, TX, project were sent to USA-CERL for analysis. RCC samples from Willow Creek Dam were analyzed at the CE laboratory in Troutdale, OR.\* Results of these tests

<sup>2</sup> R. T. Kelly and J. W. Vail, "Rapid Analysis of Fresh Concrete," *Concrete* (April 1968), pp 140-145, and (May 1968), pp 206-210.

<sup>3</sup> P. A. Howdyshell, *Operations Guide: Water and Cement Content of Fresh Concrete*, Technical Report (TR) M-177/ADA022697 (U.S. Army Construction Engineering Research Laboratory [USA-CERL], September 1975).

<sup>4</sup> P. A. Howdyshell, *Revised Operations Guide for a Chemical Technique to Determine Water and Cement Content of Fresh Concrete*, TR M-212/ADA039120 (USA-CERL, April 1977).

<sup>5</sup> P. A. Howdyshell, *Corps of Engineers Concrete Quality Monitor: Operations Guide*, TR M-293/ADA102753 (USA-CERL, May 1981).

\*North Pacific Division Laboratory at which much of the laboratory work for Willow Creek Dam was done.

were used to construct calibration curves, which were compared with known water and cement contents. Drawing on the experience with these tests, modifications were proposed and tested in the laboratory. The modified methods were then field tested at Willow Creek Dam (Heppner, OR) and Truscott, TX.

#### Mode of Technology Transfer

It is recommended that the procedures verified by this study be incorporated in the Corps of Engineers *Handbook for Concrete and Cement* (U.S. Army Waterways Experiment Station, 1949-). These procedures also should be submitted for consideration as an American Society for Testing and Materials (ASTM) standard test method.

## 2 TESTING WITH SOIL CEMENT

#### Initial Tests

Samples of the soil and cement to be used for soil cement at Truscott, TX, were sent to USA-CERL. Initial analysis indicated the CE CQM could be used for these materials and that a 1-kg (2.2-lb) sample should be large enough. The calibration graph derived from the samples formed a straight line and a significant variation was seen between the calcium content in the soil compared to that in the soil cement planned for use. The soil's calcium content has a direct relationship to the accuracy obtainable by the CE CQM method. For example, if the soil calcium content is 17.0 mg percent, CE CQM test accuracy is 5 to 7 percent, which is approximately 1.02 mg percent. If this soil is used to produce a 2.0 percent lime-soil mixture ( $4.69 \text{ mg percent} = 2 \text{ percent lime}$ ) and is tested, the accuracy allowances in determining the soil's calcium content represent an error of 22 percent for determining the lime content before the test for lime is even started. This error will be compounded if the soil tested as a blank does not have a calcium content representative of the soil in the soil-lime mixture being tested. Appendix F shows a calibration graph for the initial analyses at USA-CERL, along with the initial calibration curve drawn at Truscott.

The CE CQM water test at USA-CERL indicated that the raw materials contained no chlorides. Therefore, the test should work.

#### Test Procedure Modifications

The sample size was decreased to 1 kg (2.2 lb) and taken from a larger sample sieved in the laboratory. (The sieved sample was also used for other tests.) Because the sample had been sieved previously, only the No. 100 sieve was used instead of a nest (consisting of No. 4, No. 50, and No. 100 sieves) for the sample washing process.

The 1-kg samples from Truscott were tested to form a calibration curve. The data suggested the raw materials contained a high calcium content. Further testing indicated drastic variation in the water's calcium content when taken from different places within the job site. To eliminate the effect of the water's calcium content variation introduced by the 250 ml (.07 gal) of tapwater and 30 ml (.0008 gal) of 5 percent nitric acid, distilled water was used to replace tapwater and for preparing the 5 percent nitric acid. Use of distilled water eliminated most of the calcium variation, but Truscott personnel still found some variation in the raw materials' calcium contents.

The procedure that finally eliminated most of the calcium content variation involved the testing of a blank soil sample each day. When a soil-cement test was run, the calcium reading from the blank soil sample was subtracted from the soil-cement reading. This reading was then used with a calibration graph that related cement-only content to the calcium meter reading, and cement content could thus be determined.

Truscott personnel did not require the speed provided by the CE CQM water determination and, therefore, opted for the hot-plate drying method. (Their onsite raw materials also contained large amounts of chloride.) In the hot-plate drying method, a sample is weighed, heated over a hot plate for a specific time during which the moisture evaporates, and reweighed to determine the water content.

#### Test Data and Results for Soil Cement

The amount of cement actually added to the mixer was monitored. Actual cement was compared with cement content based on the CE CQM determinations and estimated lifts, and these data were recorded. For 70 tests, the average (CQM cement/actual cement) was .999 with an SD of .094 (which includes the error associated with estimating lifts of placement). Appendix G summarizes the data collected.

### **3 TESTING WITH ROLLER-COMPACTED CONCRETE**

#### **Initial Tests**

Initial tests performed for Willow Creek at the CE laboratory in Troutdale, OR, were to evaluate the CE CQM's applicability to RCC. The tests consisted of running four hand-mixed, known cement content, 4-kg (8.8-lb) mixes and one blank mix containing all raw materials, except cement, in the proportions that would be required for a 4-kg (8.8-lb) mix. Results of these tests were used to construct a calibration curve which formed a straight line as required, since cement content is linearly related to calcium content. The calibration curve was then used to determine the cement contents of five mixes prepared by Troutdale personnel in a mixer. The CE CQM determined the cement contents with errors ranging from 1.5 to 6.1 percent (based on CE CQM cement contents divided by actual cement content). Water tests were also performed. Two of the four water tests produced large errors compared to known water contents, requiring further verification in the field. Appendix B shows these data.

A small concrete mixer was used to make several batches of concrete at Willow Creek. CE CQM results, using the calibration curve from four hand-mixed, 4-kg (8.8-lb) samples, were compared to the batches' known cement contents. The errors for cement contents determined on two of the batches were 2.9 and 4.8 percent. Three more batches of concrete were mixed to compare CE CQM results with Troutdale Laboratory's analysis. The CE CQM was run on both whole and minus-four only samples to see if there is a consistent correlation between whole and minus-four results. Troutdale laboratory analyzed only the minus-four samples. (Appendix C contains these data.)

Comparison of Troutdale and CE CQM data indicated the CE CQM produced more consistent results under these test criteria. In addition, these results indicated the CE CQM is able to determine RCC cement content.

#### **Followup Tests and Findings**

Using Type II cement and a .03 m<sup>3</sup> (1 cu ft) mixer to prepare concrete, six sets of different mix design tests (two samples of each mix) were run. Graphical comparison of the mixes' results indicated an inconsistency in known cement content versus calcium readings; i.e., there was no linear relationship between these two parameters. Therefore, the concrete's raw

materials were tested individually to determine their calcium reading contributions. With the amounts of the raw materials per mix design and their calcium readings known, calibration curve data were calculated. (For calculation examples, see Appendix D.) The calculated calibration curves' accuracy was verified by using the CE CQM on two more mixes and showing that the results coincided with calibration curve predictions.

Investigation into the inconsistency of calcium readings with cement content demonstrated the importance of good stirring in the 37.8-L (10-gal) suspension tank while sampling. During the last half of testing, the stirring motor quit functioning because a leak damaged it; this system was replaced by manual stirring in the suspension tank. Manual stirring was thought to be a simple, straightforward replacement. However, when the cement-only samples were run, the need for vigorous manual stirring became evident. Because of insufficient stirring, one of the cement-only samples tested was off by 10 percent (i.e., not linear with respect to the other tested calcium readings). It is therefore very important to provide strong enough stirring if concrete testing must continue with an inoperable electric stirring motor.\*

#### **Test Procedure Modification**

The time involved in performing the cement determination test was identified as one problem, with the process of washing fines from the concrete initially taking 15 min. Willow Creek personnel noted that a dispersant used in aggregate testing made the washing process easier and faster. (The dispersant used was Calgon™.) The usual washing method involves moving the aggregate around manually while running the recirculating water over it. The RCC required manual rubbing of the aggregate to free all of the fines.

Calgon™ is composed primarily of phosphates. Since Corning's operator manual for the calcium meter said the presence of phosphates could interfere with the CQM's fluorometric calcium determination, this company was consulted to determine what level of phosphates causes interference. The Calgon™ manufacturer was asked to supply the composition of this water softener. This information indicated that 200 g (7 oz) of Calgon™ per test sample should not affect the calcium determination.

\*Because of many problems associated with the suspension tank's leaking out the bottom around the stirring blade rod, a different washing machine design is being investigated. Meanwhile, it is suggested that a domestic type portable washing machine be used instead of the CE CQM suspension tank or that the stirring rod seal be replaced frequently.

To test Calgon's™ effect, two sets of samples were run. One set consisted of 168 g (5.9 oz) cement mixed with 37.8 L (10 gal) of water; the sample was tested, Calgon™ was added, and the sample was retested. Table 1 gives the samples and time effects tested in the first set.

In the second set, the following samples were analyzed:

**Sample Taken from 10-gal Water Containing:**

200 g (8.44 oz) cement  
200 g (8.44 oz) cement + 100 g (4.22 oz) Calgon™  
200 g (8.44 oz) cement + 200 g (8.44 oz) Calgon™

The test results verified the manufacturer's speculation that 200 g (8.44 oz) of Calgon™ in 37.8 L (10 gal) water will not affect cement determination in this method. Willow Creek's test procedure was therefore modified to add 200 g (8.44 oz) Calgon™ to the 37.8 L (10 gal) water before washing concrete samples.

Because of the cement mixed fines' strong adhesion to the aggregate, the concrete had to be cleaned manually with a wire brush. This was done by placing the test sample into a pan with approximately 3.8 L (1 gal) of the 37.8 L (10 gal) Calgon™-water and scrubbing the concrete with a brush. The 3.8 L (1 gal) of washwater and aggregate were then poured onto the sieves and the normal procedure was followed.

Another modification involved the test sample size. The large aggregate (7.6 cm [3 in.]) mandated test samples larger than 2 kg (4.4 lb). Tests at Troutdale in August 1981 indicated that 4-kg samples would minimize the major impact of sampling errors associated

with 7.6 cm (3 in.) aggregate mixes. Although not used at Willow Creek, a suggested modification is to retain the plus-four aggregate and use this in the ANSI/ASTM C 127-91 method<sup>6</sup> to correct for 7.6 cm (3 in.) aggregate bias. (The sampling error involved in the plus 3.8 cm (1.5 in.) aggregate bias probably was a major contributor to error in the CE CQM cement content determinations.)

**Test Data and Results for RCC**

The CE CQM cement content data were compared with theoretical cement contents in the mixes and to strengths of the small cylinder breaks (15 by 30 cm [6 by 12 in.] compressive cylinder). The CE CQM cement/theoretical cement averages ranged from .97 with a standard deviation (SD) of .24 for an 80 + 32 mix to an average of .92 with an SD of .10 for a 330 + 130 mix (see Appendix E). Better accuracy could be attained if the plus-four aggregate is retained during the washing procedure, weighed, and results of the cement determination corrected for aggregate biasness using ANSI/ASTM C 127.

The CE CQM water test was performed, as with conventional concrete, on samples 2 kg (4.4 lb) or greater in mass. The value of these data is minimal, though, as water is added onsite just before the material is rolled; a nuclear density meter used onsite is better suited to this operation because it can test the in-place RCC's water content. It would be impractical to unearth RCC to test with the CQM.

<sup>6</sup>American Society for Testing and Materials (ASTM) Standard Method, C-127-81, *Test Method for Specific Gravity and Absorption of Coarse Aggregate* (1981).

**Table 1**

**Sample and Times Used in Testing for Water Softener Effect**

Sample Taken from 37.8-L (10-gal) Water Containing:	Time Tested from 37.8-L (10 gal) Solution	Time Tested from Beaker
168 g (5.9 oz) cement	8:40 a.m.	3:00, 3:30 p.m.
168 g (5.9 oz) cement + 100 g (4.22 oz) Calgon™	9:05 a.m.	9:10 a.m.
168 g (5.9 oz) cement + 200 g (8.44 oz) Calgon™	9:15 a.m.	3:30 p.m.
168 g (5.9 oz) cement + 200 g (8.44 oz) Calgon™	9:40 a.m.	9:45 a.m.
168 g (5.9 oz) cement + 200 g (8.44 oz) Calgon™	10:05 a.m.	10:10 a.m.

The CE CQM was used for mixer performance testing and proved to be an essential tool for establishing required mix times and evaluating various batching methods. It allows the whole mix to be analyzed rather than just minus-four contents, and can be done in 20 min.

## 4 CONCLUSIONS

The CE CQM has been tested for use in determining water and cement contents of soil cement and RCC. It is concluded that:

1. The CE CQM's ability to accurately and reliably work for unconventional concrete can be determined by running several combinations of the raw materials. Results of these tests should validate a calibration curve drawn from data on those materials. If there are discrepancies, the reason(s) should be determined. If possible, the procedure should be modified to correct the problems. If there are no discrepancies, the CQM should provide accurate results for the materials tested. Validation of the calibration chart (see Appendix A) should be done weekly, or more often, if deemed necessary.

2. Basic modifications to the CE CQM method for RCC include increasing the sample size to 4 kg (8.8 lb), adding 200 g (4.22 oz) Calgon<sup>TM</sup> to the 37.8 L (10 gal) of suspension washwater, using a wire brush to aid the washing process, and possibly using ANSI/ASTM method C 127 to correct for plus 3.8-cm (1.5-in.) aggregate bias. Also, a different design of suspension tank or a domestic portable washing machine should be used instead of the CE CQM suspension tank.

3. True potential accuracy of the CE CQM with RCC is difficult to establish using the Willow Creek data, because the comparison was for theoretical mix design amounts versus CQM rather than the actual contents versus CQM. Also, the cement contents determined could be biased greatly by one piece more or less of 7.6-cm (3-in.) aggregate.

4. The CE CQM proved to be essential for mixer performance testing with RCC. It allowed the entire sample to be analyzed, as opposed to only minus-four samples; it also allowed rapid, onsite testing rather than the full week required to receive results from the precipitate chemical process for cement content evaluation. The CE CQM was used to establish mix times and to evaluate various batching methods for RCC.

5. Basic modifications to the CE CQM cement content method for soil cement include decreasing the sample size to 1 kg (2.2 lb) and using only a No. 100 sieve for washing the sample. If the raw materials vary in calcium content, as they did at Truscott, the method must be further modified.

6. Cement content accuracy using the CE CQM on soil cement should be within 5 to 7 percent (the Truscott data gave a 9 percent standard deviation, but this included the error associated with estimating lifts of placement).

7. Field tests of the CE CQM with modifications described above verified this method's accuracy and feasibility for determining water and cement contents of soil cement and RCC. However, because water is added just before rolling the RCC, a nuclear density meter may be more practical for onsite testing of RCC water content.

## **APPENDIX A: EQUIPMENT AND OPERATIONS FOR CE CQM**

### **Equipment**

Tables A1 and A2 list the kinds of equipment (and their costs) needed for the CE CQM water and cement content tests (also see Figures A1 and A2). In general, this equipment is the recommended minimum needed for the CE CQM analysis. Several items can be replaced by other pieces of equipment that perform the same function. For example, the cement suspension tank (item 5, Table A2) can be replaced by the commercially produced washing machine that was specified for the original KV and USA-CERL/KV methods. A cement suspension tank was chosen for the Generation 3 system because it is smaller and more easily portable than the commercial washing machine. The triple-beam scale (item 1, Tables A1 and A2) can be replaced by a more versatile, rugged (and expensive) digital scale. Also, if the CE CQM is to be used extensively at the same site, it probably would be cost-effective to obtain or make some type of end-over-end mixer similar to those specified for the original KV and USA-CERL/KV methods to mechanically mix the water test sample in the wide-mouth jar.

Approximate 1983 costs for the items listed in Tables A1 and A2 are \$2250 and \$5384, respectively. Excluding items 1, 2, 3, and 9b in Table A2 (which are duplicates of items listed in Table A1), the total equipment cost for the CE CQM system is about \$7400.

### **Transportation and Field Operation Requirements**

The CE CQM is easy to transport, simple to set up and take down, self-contained, and can be operated in a variety of environments. All equipment can be carried in a car or pickup truck in a ready-to-use condition; this equipment does not have to be crated or packed with special shock isolation. For long-haul commercial transportation, all equipment can be crated in cardboard boxes small and light enough to ship as either excess baggage on most commercial airlines or by U.S. Parcel Post. (The crates and foam liners in which the calcium analyzer, chloride meter, and scales are packed when received from the manufacturer can be saved and used when transporting the equipment commercially.) The centrifuge and cement suspension tank are rugged mechanical items that do not require special packaging or shock isolation.

Except for the calcium meter and the cement suspension tank, all crated equipment needs only to be uncrated when it arrives in the field with electrical lines connected and hooked to a 1.5-kW (or less) source of 115-V a.c. power. In addition, an ethylene-glycol (bis) tetraacetic acid (EGTA) reagent bottle must be placed in a side compartment of the calcium meter. After the reagent bottle tubing is connected, a purge cycle must be run to remove air pockets from it. It takes about 5 min to install and purge the EGTA system.

The cement suspension tank is normally shipped in two crates: the upper section is a 37.8-L (10-gal) polypropylene tank; the lower section is a base stand which has a water pump and stirrer motor. The tank is set up by (1) placing it on the base stand, (2) attaching the tube connecting the water pump to the tank, and (3) connecting the Jiffy stirrer blade to the stirrer motor through the watertight bushing. It usually takes about 1 hr to uncrate (or crate) and set up (or take down) all the CE CQM equipment.

The CE CQM system, including prepared reagents supplied by the equipment manufacturer, is completely self-contained except for the 110 to 115 V of a.c. current (less than 1.5 kW required), tapwater, salt solution, and a 5 percent nitric acid solution. If not available locally, the current can be supplied by a small gasoline-driven generator, and the tapwater by a suitably sized storage tank. The salt solution can be made from table salt. Thus, the only material or equipment not normally available locally (or not shipped as part of the test system) is the nitric acid. Nitric acid is classified by the U.S. Department of Transportation as an oxidizer requiring an oxidizer-corrosive label and subject to transportation restrictions. These restrictions include special packaging and forbid shipment on passenger-carrying aircraft and railcars.

All CE CQM equipment, including the calcium analyzer and chloride meter, is rugged and reliable enough to operate in any indoor or outdoor environment in which concrete is normally placed.

### **Water Content Test**

#### **Reagents**

The reagents needed to conduct the water content test are:

1. Sodium chloride (NaCl) solution (about 0.5 normal [N] in tapwater).
2. Acid buffer solution.

The NaCl solution is made by dissolving 292 ( $\pm 3$ ) g ( $10.3 \pm .01$  oz) of dry NaCl in tapwater and diluting to 10 L (2.6 gal).\* Each water test uses 250 ml (.07 gal) of the 0.5 N NaCl solution; thus, a 10-L (2.6 gal) supply is enough for 40 water tests.

The acid buffer solution is a prepared reagent for the Corning 920M chloride meter. Replacements are available from Corning distributors. The reagent bottles, as shipped, contain 475 ml (.0125 gal) of solution. This is enough to load the meter's sample beaker 25 to 30 times. Each loading is good for five to eight chloride readings. If each water content test requires two to three individual readings, the 475 ml (.0125 gal) should be enough for 60 to 100 water content tests.

#### Procedure

The CQM water content test consists of adding 250 ml (.07 gal) of a 0.5 N NaCl solution to a 2-kg (4.4-lb) concrete sample, intermixing the two, and determining the chloride concentration of the intermixed supernatant salt solution using the Corning 920M chloride meter. If the concrete contains chlorides from other sources, both an actual and a blank sample (250 ml [0.7 gal] of distilled water added to a 2-kg [4.4-lb] concrete sample) must be used.

Steps for the CE CQM water content test are described below:

Step 1. Obtain a 12-to 15-kg (26.4- to 33-lb) sample of fresh concrete, mix the sample to ensure homogeneity, and weigh out two subsamples of at least 2000 ( $\pm 200$ ) g ( $4.4 \pm .44$  lb) each.\*\* Record the exact weight of each subsample to the nearest gram. place one 2-kg (4.4-lb) subsample in a wide-mouth jar; then, using a volumetric flask, add 250 ml (.07 gal) of distilled water. Secure the lid on the jar. This is the blank sample required for estimating chlorides in the concrete itself.

Step 2. Place the second 2-kg (4.4-lb) sample in another wide-mouth jar, add 250 ml (.07 gal) of 0.5 N NaCl solution, and secure the lid.

Step 3. Turn the two jars end-over-end, either by hand or in an end-over-end mixer. At least 75 complete

\*Dry NaCl crystals dissolve slowly and mechanical agitation is recommended to ensure that the crystals completely dissolve.

\*\*The reason for the wide range of sample weight is to prevent biasing the mortar/aggregate ratio of the sample by adjusting the sample size.

revolutions are recommended if the jars are turned by hand; if turned by a 40- to 60-rpm mixer, at least 2 min are recommended.\*

Step 4. After mixing, unfasten the lids and pour the water-cement slurry from the blank sample and the NaCl solution-cement slurry from the actual sample into the centrifuge tubes. Place the tubes in the centrifuge and run at 2000 to 3000 rpm for 3 to 4 min.

Step 5. Prepare the chloride meter for analysis by (a) placing the sample selector toggle switch on  $100 \mu\text{l}$  ( $2.6 \times 10^{-4}$  gal) and switching the on/off switch to on, (b) placing 15 to 17 ml (3.9 to  $4.4 \times 10^{-4}$  gal) of acid buffer solution in the meter's 20-ml ( $5.2 \times 10^{-4}$  gal) beaker, (c) placing the beaker on the stand, (d) lowering the silver electrodes, and (e) beginning the conditioning cycle by pressing the conditioning switch. (This step is required only at the start of each day or when the buffer solution sign indicates that it needs changing -about every five to eight readings.)

Step 6. Determine the chloride strength of the blank sample by pipetting  $20 \mu\text{l}^{**}$  ( $2.6 \times 10^{-5}$  gal) of the blank sample, using an Eppendorf, into the meter's 20-ml ( $5.2 \times 10^{-4}$  gal) beaker. Press the titration switch. Record the results and repeat the test to ensure reproducibility (Bl in Equation A1). If the meter's blank light is on, no chlorides are present.

Step 7. Determine the chloride strength of the actual sample by pipetting  $20 \mu\text{l}^{**}$  ( $2.6 \times 10^{-5}$  gal) of the actual sample, using an Eppendorf, into the meter's 20-ml ( $5.2 \times 10^{-4}$  gal) beaker. Press the titration switch. Record the result and repeat the test to ensure reproducibility to  $\pm 1$  percent (Sa in Equation A1).

Step 8. Determine the chloride strength of the 0.5 N NaCl solution by pipetting  $20 \mu\text{l}^{**}$  ( $2.6 \times 10^{-5}$  gal) of the .5 N NaCl solution, using an Eppendorf, into the 20-ml ( $5.2 \times 10^{-4}$  gal) beaker. Press the titration switch. Record the results and repeat the test to ensure reproducibility to  $\pm 1$  percent (Std). Water content is calculated as follows:

$$\text{Water content (ml)} = 250 \left[ \frac{\frac{\text{Std}}{\text{SaWt}} - 1}{\frac{\text{Sa} - \text{Bl}}{\text{BlWt}}} \right] \quad [\text{Eq A1}]$$

\*Under no condition should the jars be turned so rapidly that the centrifugal force exceeds gravitational forces; excess speeds prevent the salt solution and distilled water from completely mixing with the concrete samples.

\*\* $100 \mu\text{l}$  samples may be used. (The  $20 \mu\text{l}$  samples give around 100 for the NaCl chloride meter reading.)

**Table A1**  
**Water Test Cost Breakdown**

Item	Title	Quantity	Description	Source	Cost (\$)	
					Per Unit	Total
1	Scale	1	Triple-beam scale (2600 g capacity, 1 g sensitivity)	Laboratory equipment supplier	143	143
2	Hand scoop	1	Square-mouth scoop; bowl dimensions are 3 in. (76 mm) wide by 8 in. (203 mm); 1 mg; cast aluminum	Equipment supplies, concrete & soil testing	10	10
3	Sample tub	1	5 qt (4.7 L) polyethylene tub	Domestic food freezer goods supplier	1	1
4	Wide-mouth jar	2	1/2 gal (1.9 L) polyethylene wide-mouth jar with screw closure and lid	Laboratory equipment supplier	6	6
5	Centrifuge	1	Variable speed, 4-place centrifuge for 15 ml tube	Same as Item #1	244	244
6	Centrifuge tube	2	Disposable, 15 ml, polystyrene centrifuge tubes (2 per test purchased in cases of 1000)	Same as Item #1	.12/pair	62/1000
7	Eppendorf pipet (20 $\mu$ l)	1	Tip ejector fixed volume, pipet (20 $\mu$ l) capacity	Same as Item #1	90	90
8	Disposable pipet	3	Disposable tips for 20 $\mu$ l Eppendorf pipets; purchased in case lots of 1000	Same as Item #1	37/1000	37/1000
9	Chloride meter	1	Corning Model 920M chloride meter	Same as Item #1	1600	1600
10	Volumetric flask	2	Polypropylene, 250 ml cap	Same as Item #1	10	20
11	Carboy	1	Linear polyethylene, rectangular, with spigot, screw closure, 2 gal (7.6 L) capacity	Same as Item #1	35	35
12	Beakers	1	Polypropylene, Griffin low-form graduated, 250 ml capacity (sold in case lots of 6)	Same as Item #1	2	12
					Total	2260



**Figure A1.** Equipment for CQM water test.

**Table A2**  
**Cement Test Cost Breakdown**

Item	Title	Quantity	Description	Source	Cost (\$)	
					Per Unit	Total
1	Scale	1	Triple-beam scale (2600 g capacity, 1 g sensitivity)	Laboratory equipment	143	143
2	Hand scoop	1	Square-mouth scoop bowl; 3 in. (76 mm) wide by 8 in. (203 mm) long; cast aluminum	Equipment supplier for concrete and soil testing	10	10
3	Sample tub	1	5 qt (4.7 L) polyethylene tub	Domestic food freezer goods supplier	1	1
4	Specimen tub	1	2 qt (1.9 L) polyethylene tub	Same as Item #3	1	1
5	Cement suspension tank	1	Polypropylene, 10 gal (37 L) "Nalgene" tank including recirculating pump and hose, 1/20 hp DC motor with an AC/DC controller for use on 115/120-Volt AC lines, watertight bushing, and Jiffy mixing blade coupled through universal joint to 1/12 hp stirrer motor. Including cutout ring to hold 12 in. (304 mm) diameter sieves.	Tank, hoses, motors, etc. purchased from laboratory equipment supplier (locally fabricated)	1050	1050
6	Sieve nest	1	Standard stainless steel 12 in. dia (304 mm)	Same as Item #2	88	88
	No. 4		4760 micron openings		85	85
	No. 50		297 micron openings		85	85
	No. 100		149 micron openings		85	85
7	Magnetic stirrer & stirring rod		Variable speed magnetic stirrer & non-stick coated stirring rod	Same as Item #1	94	94
8	Syringe type pipet	1	"Varipet," syringe-type variable volume transfer pipet, 30 ml capacity	Same as Item #1	64	64
9	Eppendorf pipet		Tip ejector, fixed volume, pipet	Same as Item #1	60	60
	20 $\mu$ l capacity	1			90	90
	100 $\mu$ l capacity	1			90	90
10	Disposable pipet tips		Disposable tips for 20 and 100 $\mu$ l Eppendorf pipets, purchased in case lots of 1000	Same as Item #1	37/1000	37/1000
11	Flask			Same as Item #1		
	Erlenmeyer (a)	1	Polycarbonate, 500 ml capacity		6	6
	volumetric (b)	1	Polypropylene, 250 ml capacity		10	10
12	Calcium analyzer	1	Corning Model 940 calcium analyzer	Same as Item #1	3495	3495
13	Carboy	1	Linear polyethylene, rectangular, with spigot screw closure, 2 gal (7.6 L) capacity	Same as Item #1	35	35
					Total	5529

CERL - CQM

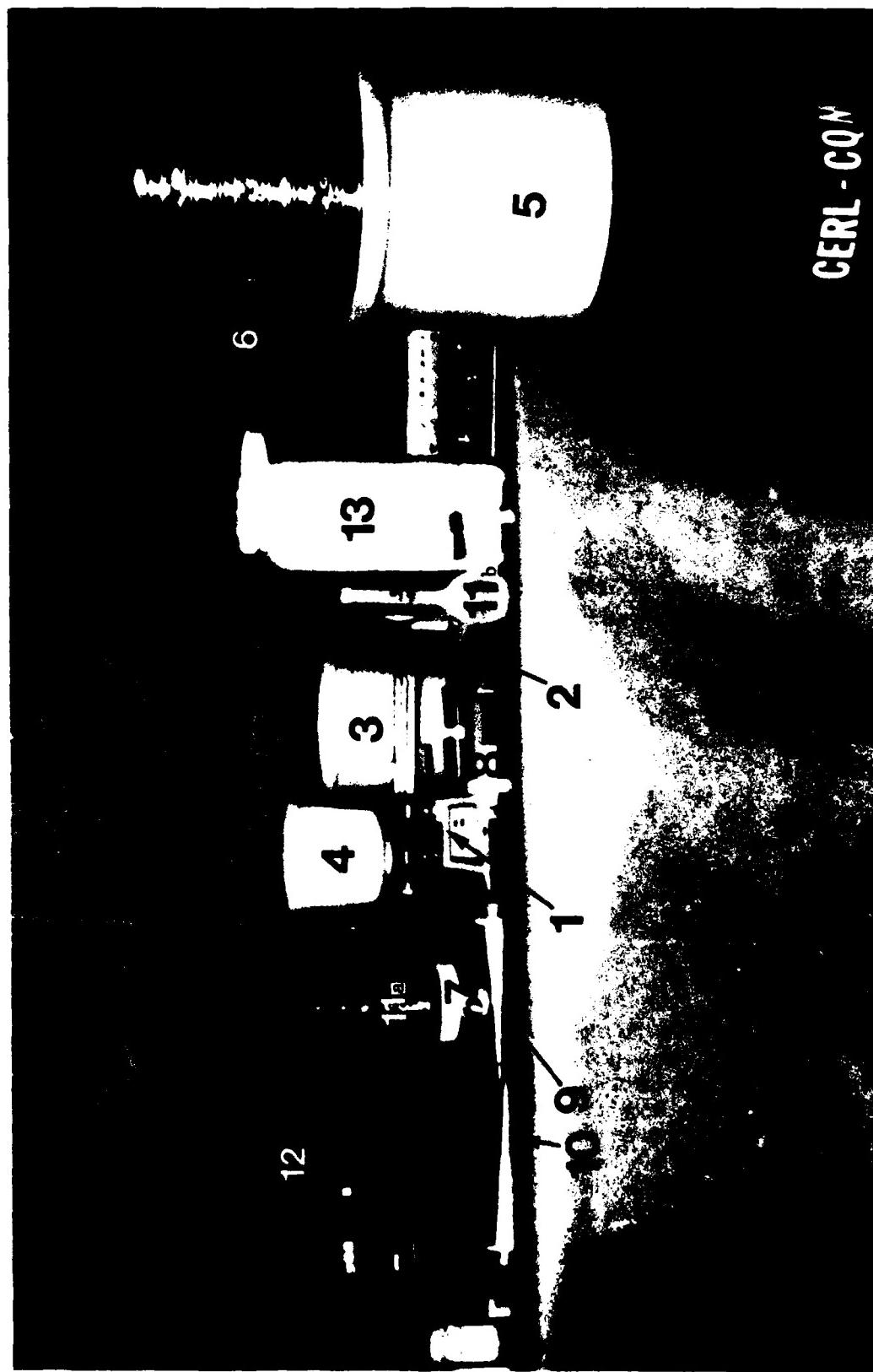


Figure A2. Equipment for CQM cement test.

where:

$Std$  = relative chloride strength of the 0.5 N NaCl solution (Step 8) (meq/L)

$Sa$  = relative chloride strength of the sample solution (Step 7) (meq/L)

$Bl$  = relative chloride strength of the blank solution (Step 6) (meq/L)

$SaWt$  = weight of sample (nearest gram)

$BlWt$  = weight of blank (nearest gram).

If the concrete being tested does not contain chlorides (i.e., if the chloride strength of the blank sample equals zero), the blank sample tests can be ended.

#### Cement Content Test

##### Reagents

The reagents needed to conduct the cement content test are:

1. A 5 percent nitric acid solution made by adding 5 ml ( $1.3 \times 10^{-4}$  gal) of nitric acid (specific gravity = 1.42) to 95 ml ( $2.5 \times 10^{-3}$  gal) of tapwater.
2. Tapwater.
3. EGTA solution.
4. Calcein indicator solution.
5. Calcium standard solution.
6. Potassium hydroxide (1.0 N).

Reagents 3 through 6 are preprepared and are available from Corning (produced specifically for Corning's 940 calcium analyzer). Replacements are available from Corning distributors. Reagent 4, the calcein indicator, is shipped in a powder form; each pre-packaged sample of calcein powder is dissolved in 10 ml ( $2.5 \times 10^{-4}$  gal) of the calcium standard solution. The reconstituted calcein solution has a shelf life of 4 to 6 weeks and should be replaced accordingly.

##### Calibration Requirements

Before conducting the cement content test, the procedure must be calibrated for the calcium in the cement and concrete raw materials. This is done by running the standard cement content test. To determine

the aggregate blank calibration value, Step 2 of the cement test procedure is excluded, and the aggregate proportions and additives present in a 2-kg (4.4-lb) concrete sample are obtained as the "2-kg (4.4 lb) sample." In Step 10, a 100- $\mu$ l ( $2.6 \times 10^{-4}$  gal) sample is analyzed in the calcium meter.

For the concrete calibration test, a 2-kg (4.4-lb) sample of concrete is hand-mixed using the materials and mix proportions of the concrete to be tested (Steps 1 through 10), and the results are recorded.\*

The cement calibration curve is a linear plot of cement content (g) versus the calcium analyzer reading (mg %), with zero cement being the aggregate blank calibration result (mg %) divided by 5. The weight of the cement in the 2-kg (4.4-lb) concrete calibration sample and its calcium analyzer reading is the other set of coordinates. Figure A3 shows a typical calibration curve.

The concrete calibration test must be repeated each time the cement, aggregate source or type, or water source used to produce the concrete changes (or on a weekly basis if the aggregate and cement sources and type do not change).

##### Procedure

The CE CQM cement content test is based on the following assumptions:

1. Cement of a given type from a given source is uniform in calcium content; the aggregates either do not contain calcium or are uniform in calcium content for that proportion of the aggregates that pass the finest sieve over the cement suspension tank.
2. When agitated, cement can be dispersed uniformly and suspended in water so that a representative sample can be obtained.
3. Stirring without external heat will produce a quantitative solution of cement in nitric acid.
4. The calcium content of the cement solution can be determined by titration with the Corning 940 calcium analyzer.

\*The sieve arrangement used in the calibration procedure should be consistent with that used in the test procedure. That is, if only the No. 4 and 50 sieves are used to calibrate the cement test, the same arrangement should be used during testing. If the No. 4, 50, and 100 sieves are used, they should be used for both calibrating and testing.

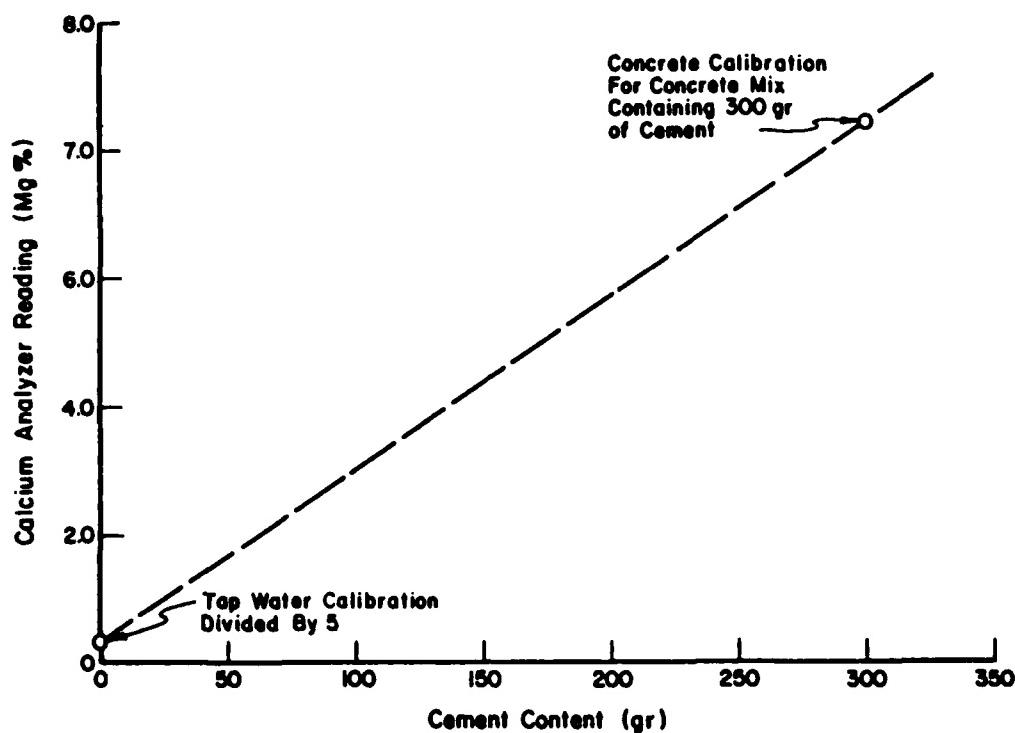


Figure A3. Typical cement calibration curve.

Steps for the cement content test are described below:

Step 1. Fill the cement suspension tank with tap-water to the 37.8-L (10-gal) mark on the side of the tank. Place the nested sieves on the tank and turn on the tank's agitator.\*

Step 2. Obtain the 12- to 15-kg (26.4- to 33-lb) concrete sample, mix the sample to ensure homogeneity (or remix, if used in conjunction with the water test), and weigh out 2000 ( $\pm 200$ ) g ( $4.4 \pm .44$  lb) of fresh concrete. Record the weight to the nearest gram.

Step 3. Transfer the 2000-g (4.4-lb) sample to the sieves over the tank. Turn on the tank's recirculating pump and wash the residue from the 2000-g (4.4-lb) sample container into the tank using the water jet from the recirculating pump hose.

\*If calcareous fines are present, it is recommended that a No. 100 sieve be nested below the No. 50. The combination of sieves used for calibration and cement content testing must be consistent.

Step 4. Wash the plus-four aggregate carefully using the water jet from the recirculating pump hose. After all the cement has been washed from the aggregate retained on the No. 4 sieve (this takes about 1 to 1.5 min), remove the No. 4 sieve.

Step 5. Wash the aggregate retained on the No. 50 sieve until all cement has been washed from the aggregate (this takes about 1 to 1.5 min). Remove the No. 5 sieve.\*

Step 6. Obtain a representative sample of the cement suspension in the tank using the 30-ml ( $7.9 \times 10^{-4}$  gal) syringe pipet. Place the suspended material in a 500-ml (.013-gal) Erlenmeyer flask. Refill the syringe pipet with 5 percent nitric acid, and add the acid solution to the contents of the Erlenmeyer flask. While discharging the acid solution from the syringe pipet, shake the pipet occasionally to ensure that all

\*When both the No. 50 and 100 sieves are used, the aggregate retained on the No. 100 sieve should be washed an additional 1 to 1.5 min after the No. 50 sieve has been removed. Remove the No. 100 sieve when complete.

that settled out when the sample was taken has dissolved and is flushed with the acid solution. Use a volumetric flask to add 250 ml (.07 gal) of tapwater to the Erlenmeyer flask.

**Step 7.** Put a magnetic stirring bar in the Erlenmeyer flask and place it on a magnetic stirrer. Turn on the stirring motor and check to see that stirring has begun.

**Step 8.** Prepare the calcium analyzer by switching the power on and filling the cuvette to the mark with 1.0 N potassium hydroxide; then add 100  $\mu$ l ( $2.6 \times 10^{-5}$  gal) (Eppendorf) of reconstituted calcein reagent. Put the cuvette in the analyzer, and add 100  $\mu$ l ( $2.6 \times 10^{-5}$  gal) (Eppendorf) of calcium standard solution, and push the titration button to condition the cuvette for analysis (This step is required only after the cuvette is filled with new potassium hydroxide solution. A single cuvette filling is enough for 15 to 20 readings.)

**Step 9.** Begin the analysis by placing the meq/mg% toggle switch on mg% and adding 100  $\mu$ l ( $2.6 \times 10^{-5}$  gal) (Eppendorf) of the calcium standard to the cuvette. Press the titration button. Record the result and repeat the test by adding another 100- $\mu$ l ( $2.6 \times 10^{-5}$  gal) sample. Keep repeating until consecutive results are less than 1.5 percent apart. Push the calibration button and run an additional 100- $\mu$ l ( $2.6 \times 10^{-5}$  gal) sample of the calcium standard to ensure that the calcium standard readout value is 10 ( $\pm 0.1$ ) mg%.

**Step 10.** Determine the strength of the cement solution in the Erlenmeyer flask by analyzing a 20- $\mu$ l ( $5.2 \times 10^{-5}$  gal) sample in the calcium analyzer. Repeat this test until all values are less than 1.5 per-

cent apart. Determine the cement content by referring to the calibration graph.

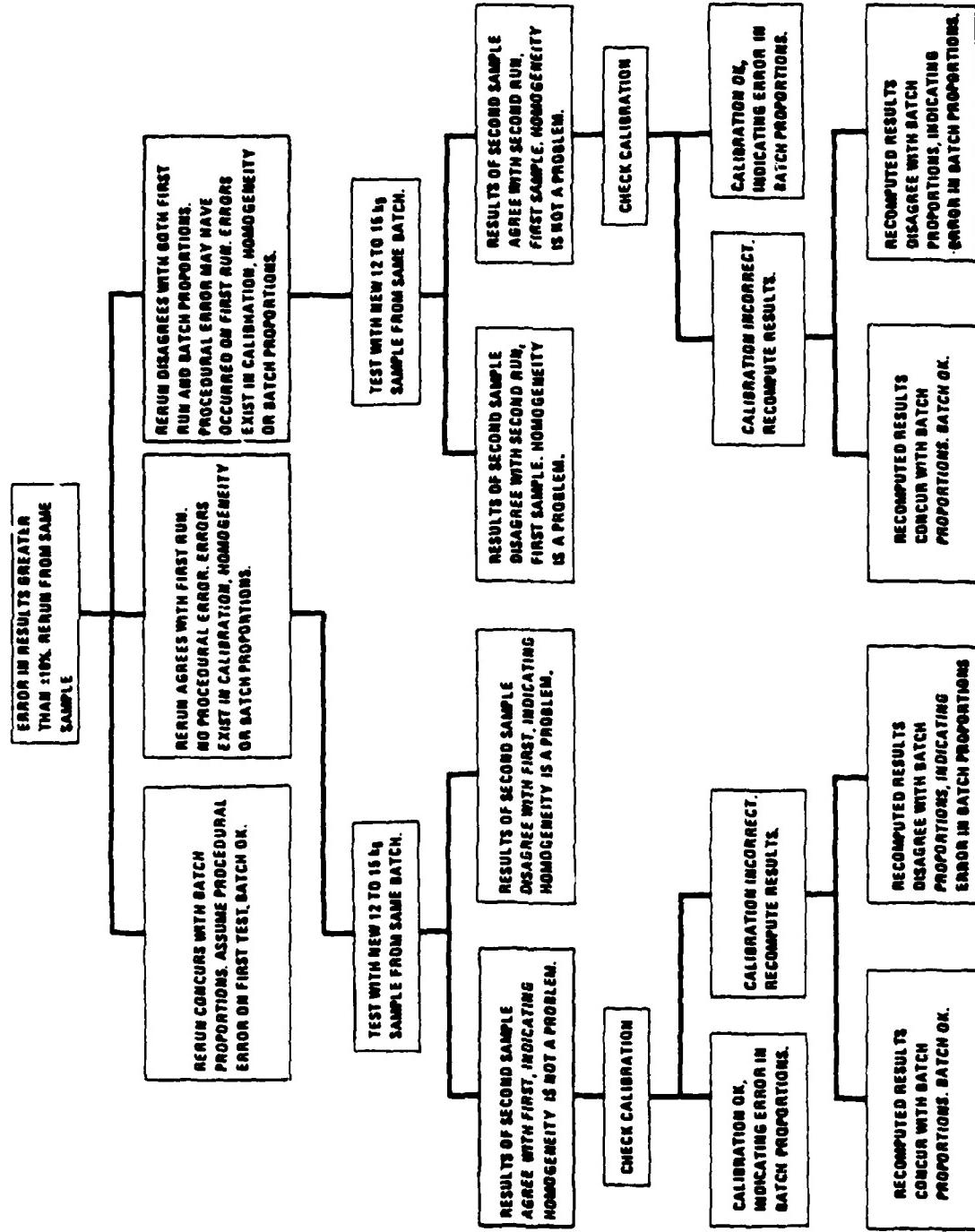
#### Comparison With Mix Design Values

The water and cement contents determined by the CE CQM method should be compared with the batch proportion values. If the CE CQM and batch proportion values vary by less than 10 percent, it is assumed that the CE CQM system is operating properly, that the concrete batch is homogeneous, and that the batched proportion values are correct. If the results vary by more than 10 percent, a second complete CE CQM test should be run. The 2-kg (4.4-lb) test samples for the rerun should be taken from the original 12- to 15-kg (26.4- to 33-lb) sample collected for the initial runs. Extreme care should be exercised on the reruns to ensure no procedural errors are made. If the second test agrees closely with the batch proportion values, it should be assumed that it is correct and that the initial test was in error. If the second test is significantly different from both the batch proportion and first test results, or if the second test agrees closely with the first, one of three things has occurred:

1. The concrete sample is not representative of the bulk (indicating poor mixer efficiency and nonhomogeneity).
2. The batch is not the same as indicated by the batch proportions.
3. The CE CQM system is incorrectly calibrated.

Figure A4 shows a series of analytical steps for determining which of these three problems has occurred. It is recommended that an inquiry be made as to any changes or problems that may have occurred at the batch plant.

## PROCEDURE FOR CHECKING CAUSE OF ERROR IN RESULTS



**Figure A4.** Procedure for checking cause of error in results.

**APPENDIX B:**  
**TROUTDALE LABORATORY PRELIMINARY RCC TESTING—**  
**AUGUST 1981**

**Cement Analysis**

Data from test samples obtained from mixer:

**80 + 32 Mix Design**

(uncorrected for coarse aggregate variance)

1. Sample wt = 4.816 kg

Ca++ meter = 11.005

From graph cement Content = 94 g/4-kg sample

$$\frac{94 \text{ g}}{4816 \text{ g}} = 1.95\%$$

Actual % = 1.98

% off = 1.5

2. Sample wt = 4.767 kg

Ca++ meter = 11.54

From graph cement content = 100 g/4-kg sample

$$\frac{100 \text{ g}}{4767 \text{ g}} = 2.10\%$$

Actual % = 1.98

% off = 6.1

3. Sample wt = 4.026 kg

Ca++ meter = 5.69

From graph cement content = 37.8 g/4-kg sample

$$\frac{37.8 \text{ g}}{4026 \text{ g}} = .94\%$$

Actual % = 1.00

% off = 6.0

4. Sample wt = 4.083 kg

Ca++ meter = 9.62

From graph cement content = 79.8 g/4-kg sample

$$\frac{79.8 \text{ g}}{4083 \text{ g}} = 1.95\%$$

Actual % = 1.98

% off = 1.5

**175 + 80 mix**

5. Sample wt = 4.008 kg

Ca++ meter = 18.3 g

From graph cement content = 165 g/4-kg sample

$$\frac{165 \text{ g}}{4008 \text{ g}} = 4.12\%$$

Actual % = 4.29

% off = 4.0

**Water Analysis: Data From Test Samples Obtained From Mixer**

Actual water content for all batches was kept at 7.9/151 lb batch. (James Hinds, Chief of Troutdale's concrete section, calculated that if 80 percent of the water that would absorb during mixing did absorb, water content ~ 4.65 percent.)

(Uncorrected for coarse aggregate variance)

1. Sample wt = 3.465 kg

Cl meter = 57.5

$$\frac{250 \left( \frac{94}{57.5} - 1 \right)}{3.465 \text{ kg}} \sim 4.58\% \text{ water} \quad \% \text{ off} = 1.5$$

2. Sample wt = 3.3395 kg

Cl meter = 58.1

$$\frac{250 \left( \frac{94}{58.1} - 1 \right)}{3.3395 \text{ kg}} \sim 4.63\% \text{ water} \quad \% \text{ off} = .4$$

3. Sample wt = 2.531 kg

Cl meter = 67.7

$$\frac{250 \left( \frac{94}{67.7} - 1 \right)}{2.531 \text{ kg}} \sim 3.84\% \text{ water} \quad \% \text{ off} = 17.4$$

4. Sample wt = 3.249

Cl meter = 49.7

$$\frac{250 \left( \frac{94}{49.7} - 1 \right)}{3.249 \text{ kg}} \sim 6.86\% \text{ water} \quad \% \text{ off} = 47.5$$

**Modifications Needed/Problems Found at Troutdale**

1. Problem: As is, the sieves fill up during washing and time must be spent to clear them before the finishing rinse.

Solution: Sieve usage or procedure must be modified.

2. Problem: Small sticks, etc., float to the top during centrifuging of the water sample.

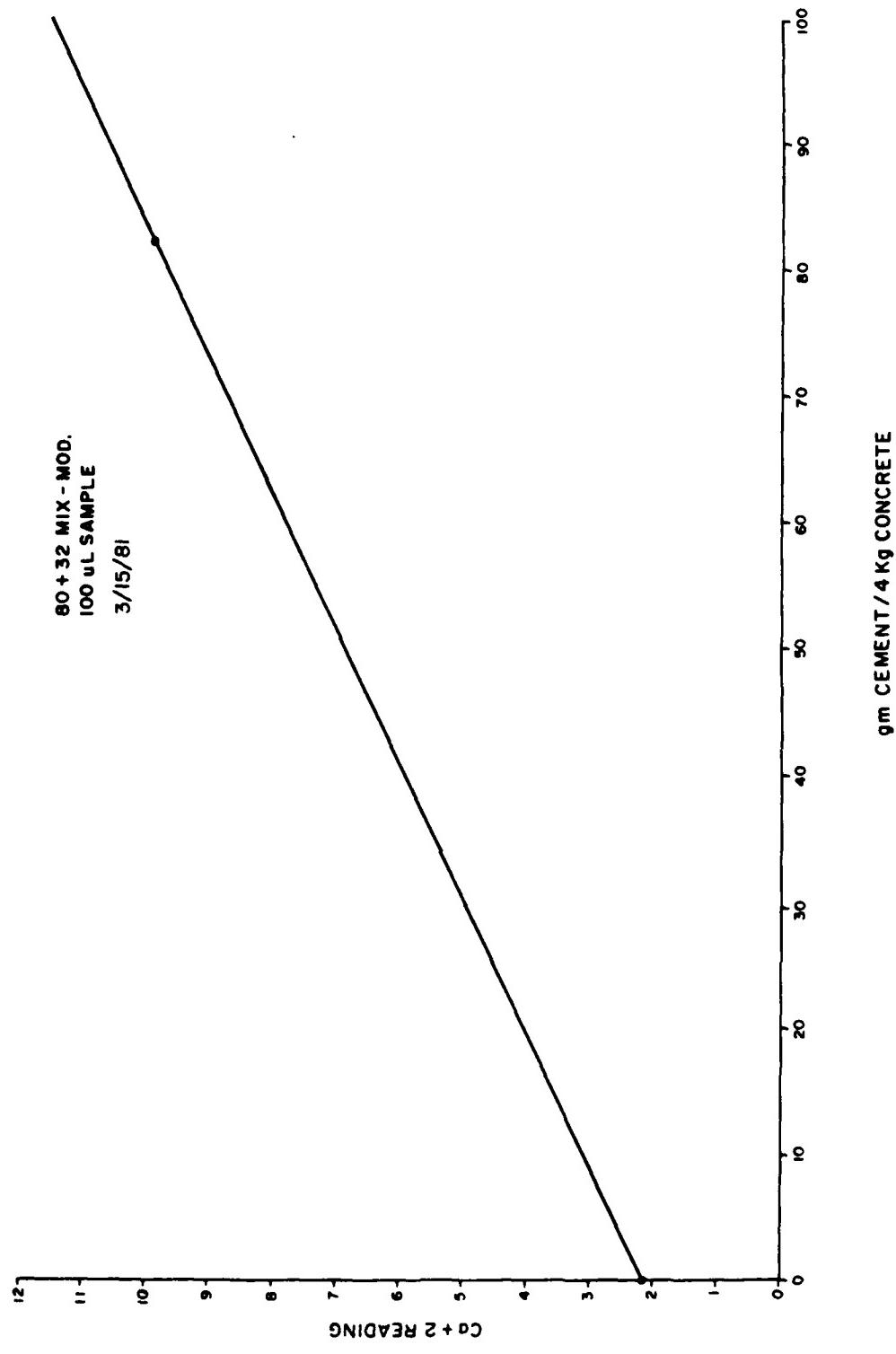
Solution: Centrifuge swabbing with possible recentrifuging might be needed.

**Data Used to Construct 80 + 32 Mix Calibration Curve**

Mix	For 4 kg	1	2	3	4	5
Cement	.0776	0	.060	.078	.090	.082
Fly Ash	.0312	.031	.031	.031	.031	.032
Water	.1940	.194	.194	.194	.194	.194
3 to 1-1/2 in.	.9316	.894	.9275	.932	.943	1.126
1-1/2 to 3/4 in.	.8636	.8575	.850	.871	.857	.860
3/4 to 0 in.	1.2520	1.253	1.2525	1.2525	1.2525	1.2525
Blend sand	.6500	.650	.6500	.6500	.6500	.650
Total wt	4.000	3.8795	3.965	4.0085	4.0175	4.1965
Ca++ meter (Sample size = 100 $\mu$ l)		2.16	7.79	9.60	10.61	9.85

**Data Used to Construct 175 + 80 Mix Calibration Curve**

Mix	For 4 kg	1	2
Cement	.1708	.171	0
Fly ash	.078	.078	.078
Water	.176	.176	.176
3 to 1-1/2 in.	.9012	.914	.921
1-1/2 to 3/4 in.	.832	.836	.853
3/4 to 0 in.	1.2124	1.213	1.2125
Blend sand	.628	.628	.628
Total wt	3.998	4.016	3.8685
<b>Sample Size</b>			
Ca++ meter	100 $\mu$ l	18.94	2.29
	20 $\mu$ l X 5	18.8	



**APPENDIX C:**  
**WILLOW CREEK DATA**

**CQM/Troutdale Laboratory Comparison**

Numbers represent the percentage cement by weight in concrete.

	CQM		Troutdale Lab		Mixed
	Whole*	-4*	-4*	-4*	Whole*
Mix #1	—	9.7%	10.8%	4.2%	
Mix #2	5.9%	9.7%	9.5%	4.2%	
Mix #3	4.9%	8.4%	7.3%	4.2%	

**Mixes Used for Calibration Chart—First Trip**

Mix design (lb)	4 kg mix (kg)	Mix 1 (kg)	Ca <sup>++</sup> meter reading (Ca <sup>++</sup> )	
			100 µl	20 µl
3 in.	.986	.962		
1-1/2 in.	.796	.777		
3/4 in.	1.680	1.640		
Sand	.315	.307		
H <sub>2</sub> O	17.6 gal X 8.33 = 146.6	.143		
Cement	<u>175</u>	.171		
Total wt	4098.6			
		Cement .176		
		3 in. .970	19.35	4.09
		1-1/2 in. .800	19.18	4.17
		3/4 in. 1.695	19.42	<u>4.13</u>
		S .315	19.25	
		H <sub>2</sub> O .150	Avg 19.30	20.65†

Mix 2 (kg)	100 µl	Mix 3 (kg)	Ca <sup>++</sup>	
			100 µl	20 µl
Cement 0		Cement .150	17.65	3.77
3 in. .930	2.75	3 in. 1.005	17.66	3.36
1-1/2 in. .780	2.71	1-1/2 in. .775	17.75	3.72
3/4 in. 1.640	2.70	3/4 in. 1.640	17.90	3.58
S .305	2.65	S .305	18.01	3.38
H <sub>2</sub> O .143	<u>2.72</u>	H <sub>2</sub> O .143	18.03	3.57
			18.03	3.39
			18.08	<u>3.54</u>
			17.95	
			Avg 17.90	17.69†

Mix 4 (kg)	100 µl	Mix 5 (kg)	Ca <sup>++</sup>	
			100 µl	20 µl
Cement .185		Cement .120		
3 in. .980	21.13	3 in. .950	14.49	3.10
1-1/2 in. .775	20.98	1-1/2 in. .780	14.47	3.06
3/4 in. 1.640	21.24	3/4 in. 1.640	14.77	3.13
S .305	21.24	S .310	14.96	3.18
H <sub>2</sub> O .143	<u>21.12</u>	H <sub>2</sub> O .143	14.93	2.93
			14.53	2.93
			Avg 14.69	3.06
				15.28†

\* "Whole" signifies a sample not sieved before testing. "-4" signifies that tested material was taken from concrete sieved through a No. 4 sieve; i.e., only material passing the No. 4 sieve was tested.

† Five times 20 µl value.

1. Test of calibration chart  
cement--3.875 -kg sample

3/10/82  
Water--2.360-kg sample

100 $\mu$ l	Sample + NaCl	NaCl Standard
20.11*	63	97
19.67	62	99
19.62	64	99
19.77	63	<u>98.3</u>
Avg 19.69	63	
$[(19.69 - 2.21) \frac{4.0}{3.875}] + 2.21^{**} = 20.25$	250 ml $[\frac{98.3}{63} - 1] = 140 \text{ ml H}_2\text{O}$	

176 g cement/4 kg concrete

Mix contained  $\frac{171 \text{ g}}{4 \text{ kg concrete}}$  : 2.9% error

$$\frac{140 \text{ ml H}_2\text{O}}{2360 \text{ g concrete}} = 5.9\% \text{ H}_2\text{O}$$

2. Test of calibration chart  
cement--3.950-kg sample

3/11/82  
Water--2.815-kg sample

100 $\mu$ l	Sample + NaCl	NaCl standard
18.73	73	97
17.60	73	99
17.92	73	98
17.99		<u>98</u>
18.32		
18.11		
Avg 18.11		
$[(18.11 - 2.21) \frac{4.0}{3.95}] + 2.21 = 18.21$	250 ml $[\frac{98}{73} - 1] = 85.6 \text{ ml H}_2\text{O}$	
157 g cement/4 kg concrete		
Mix contained $\frac{165 \text{ g}}{4 \text{ kg concrete}}$ : 4.8% error		
	$\frac{85.6 \text{ ml H}_2\text{O}}{2815 \text{ g concrete}} = 3.0\% \text{ H}_2\text{O}$	

3. Mix to compare to Troutdale cement  
determination--#1  
Minus-4 sample = 1.750 kg

3/11/82

100 $\mu$ l	386.5 g cement 4-kg sample
19.19	
19.36	
19.24	
19.32	
19.35	
Avg 19.29	
$[(19.29 - 2.21) \frac{4.0}{1.75}] + 2.21 = 41.25 :$	

\*Exclude reading: 1) was first reading which may be erratic, 2) was obviously inconsistent with others.

\*\*100  $\mu$ l of a blank sample tested produced a calcium meter reading of 2.21.

4.\* Mix to compare to Troutdale cement determination -#2

3/12/82

Whole sample = 4.950 kg

100 $\mu$ l	20 $\mu$ l
31.35	6.57
31.65	6.64
31.85	6.46
<u>32.17</u>	6.51
Avg 31.76	6.25
	6.31
	<u>6.22</u>
	6.42
$(31.76 - 2.21) \frac{4.00}{4.95} + 2.21 = 26.09$	$(32.11^{**} - 2.21) \frac{4.00}{4.95} + 2.21 = 26.37$
<u>235.5 g cement</u>	<u>238 g cement</u>
4 kg concrete	4 kg concrete

Minus-4 sample = 3.000 kg

20 $\mu$ l
6.53
6.19
6.27
<u>6.37</u>
6.34
$(31.70^{**} - 2.21) \frac{4.0}{3.0} + 2.21 = 41.53$
<u>389.5 g cement</u>
4 kg sample

5.\* Mix to compare to Troutdale cement determination -#3

3/12/82

Whole sample = 4.600 kg

100 $\mu$ l	20 $\mu$ l
25.29	4.95
25.26	4.94
25.55	5.11
25.58	5.11
<u>25.97</u>	<u>5.03</u>
25.53	5.03
$(25.53 - 2.21) \frac{4.0}{4.6} + 2.21 = 22.49$	$(25.14^{**} - 2.21) \frac{4.0}{4.6} + 2.21 = 22.15$
<u>199 g cement</u>	<u>196 g cement</u>
4 kg concrete	4 kg concrete

\*Stirred with screwdriver

\*\*20  $\mu$ l value  $\times$  5.

Minus-4 sample = 2.000 kg

100 $\mu$ l	20 $\mu$ l
18.90	3.71
19.14	3.90
19.18	3.80
19.45	3.84
<u>19.17</u>	<u>3.99</u>
	3.85

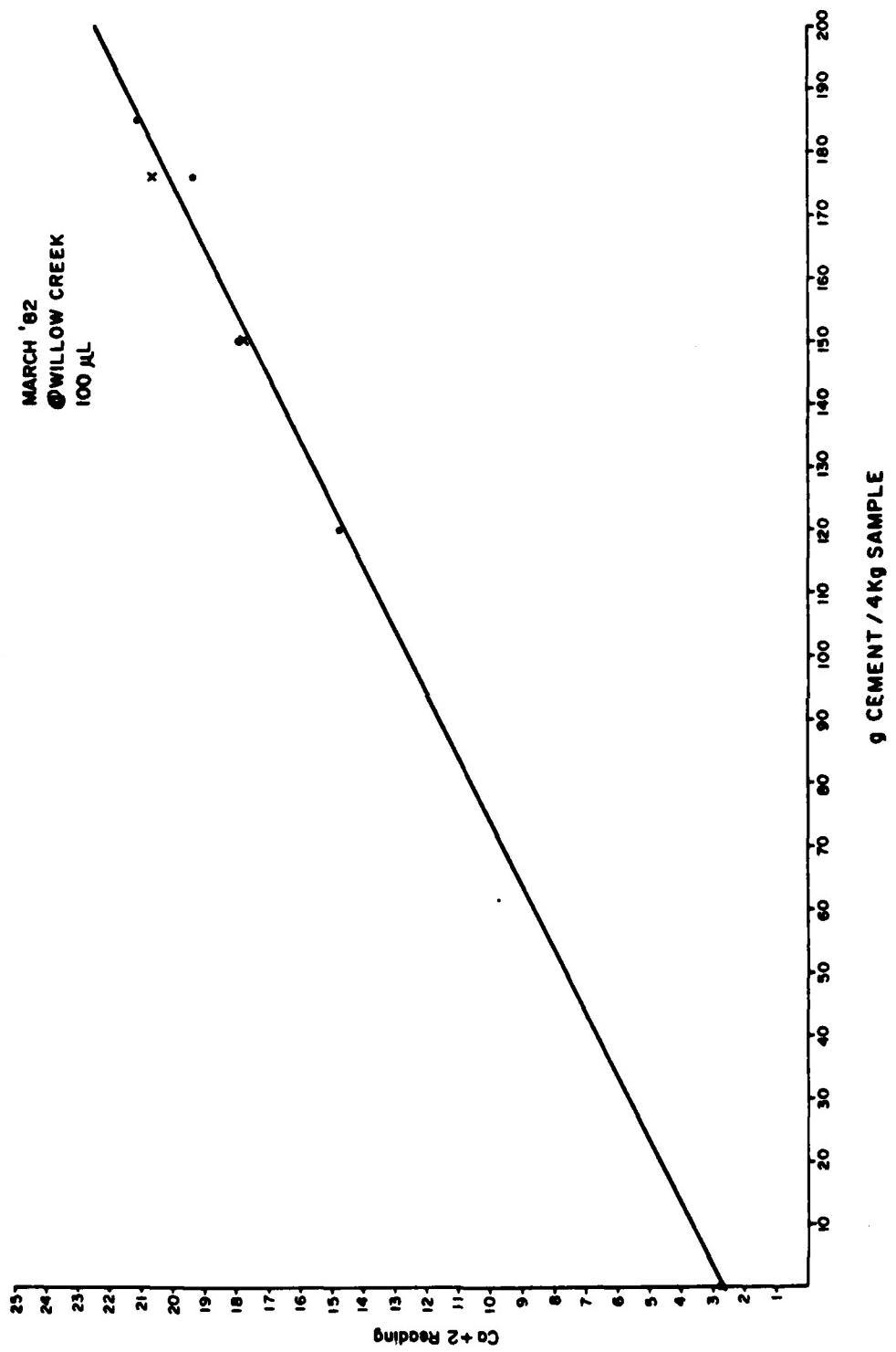
$$[(19.17 - 2.21) \frac{4.0}{2.0}] + 2.21 = 36.13$$

$$2.21 + [\frac{4}{2} (19.24 - 2.21)] = 36.27$$

$$\frac{336 \text{ g cement}}{4 \text{ kg sample}}$$

$$\frac{337.5 \text{ g cement}}{4 \text{ kg sample}}$$

$$\begin{aligned}\text{Total mix} &= (+4 \text{ mix}) + (-4 \text{ mix}) \\ 46.84 \text{ kg} &= 27.01 \text{ kg} + 19.83 \text{ kg} \\ 57.66\% &\quad 42.33\%\end{aligned}$$



**APPENDIX D:**  
**DATA FOR CALIBRATION CURVES**

**Type II Cement (for 100- $\mu$ l Samples)**

Samples run—raw materials

Cement	Cement (g)	Ca reading
	37.9	5.70
	75.8	9.575
	118.5	14.09
	165.9	19.11
	213.3	24.6
	260.7	29.63
	298.6	30.9 — wrong
	355.5	37.54

Mix	g Cement/4 kg	Calculated Ca reading
80 + 32	76.8	9.98
175 + 00	167.7	19.26
175 + 80	168.0	19.29
315 + 135	303.6	33.15
330 + 130	332.0	36.05

Fly Ash	Fly ash (g)	Ca reading
	15.1	2.24
	160.2	2.49

Mix	g Fly ash/4 kg	Calculated Ca reading
80 + 32	30.8	2.27
175 + 80	76.9	2.35
315 + 135	130.0	2.44
330 + 130	130.8	2.44

Other	Aggregate and water wt proportion*					
	pcf	4 kg	pcf	4 kg	pcf	4 kg
3 in.	45.4	1.23	—	—	—	—
1-1/2 in.	30.8	.83	60.5	1.77	121.2	3.72
3/4 in.	59.0	1.59	69.1	2.02	—	—
Sand	6.0	.16	—	—	—	—
Water	6.85	.19	7.41	0.22	9.07	0.28
	148.05		137.01		130.27	
Ca reading		2.72		2.52		3.14

\*Example for 175 + 00 mix, 3 in. =  $\frac{1228}{4174} \times 154.6$  pcf = 45.5 lb  $\approx$  45.4 for average

with other mixes and taking into account this is aggregate. These weights were then adjusted to equal 4 kg total, i.e.,

$(\frac{45.4}{148.05}) 4$  kg = 1.23 kg of 3 in. to add to other aggregate and water.

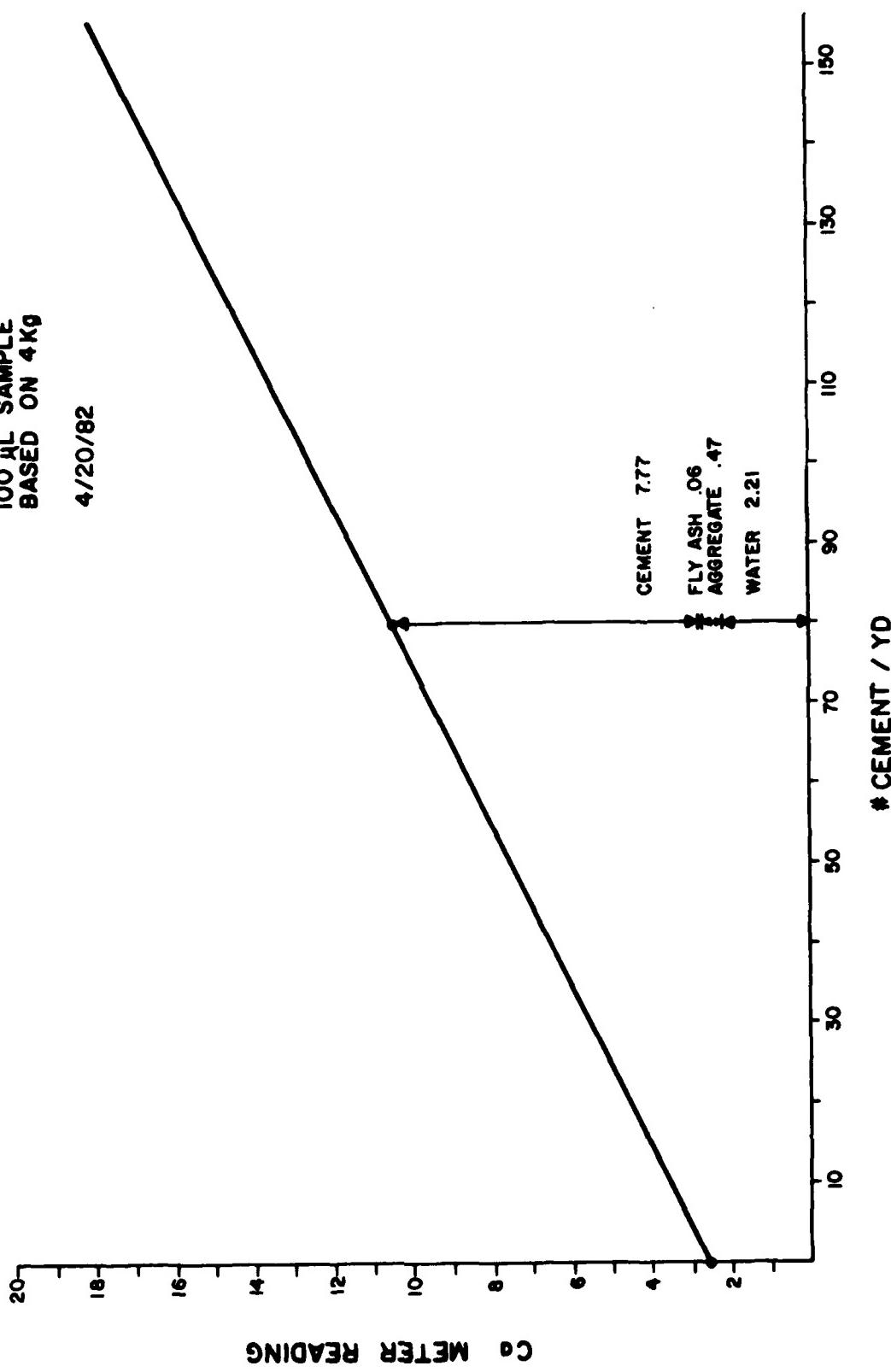
**Calculations for Mixes**

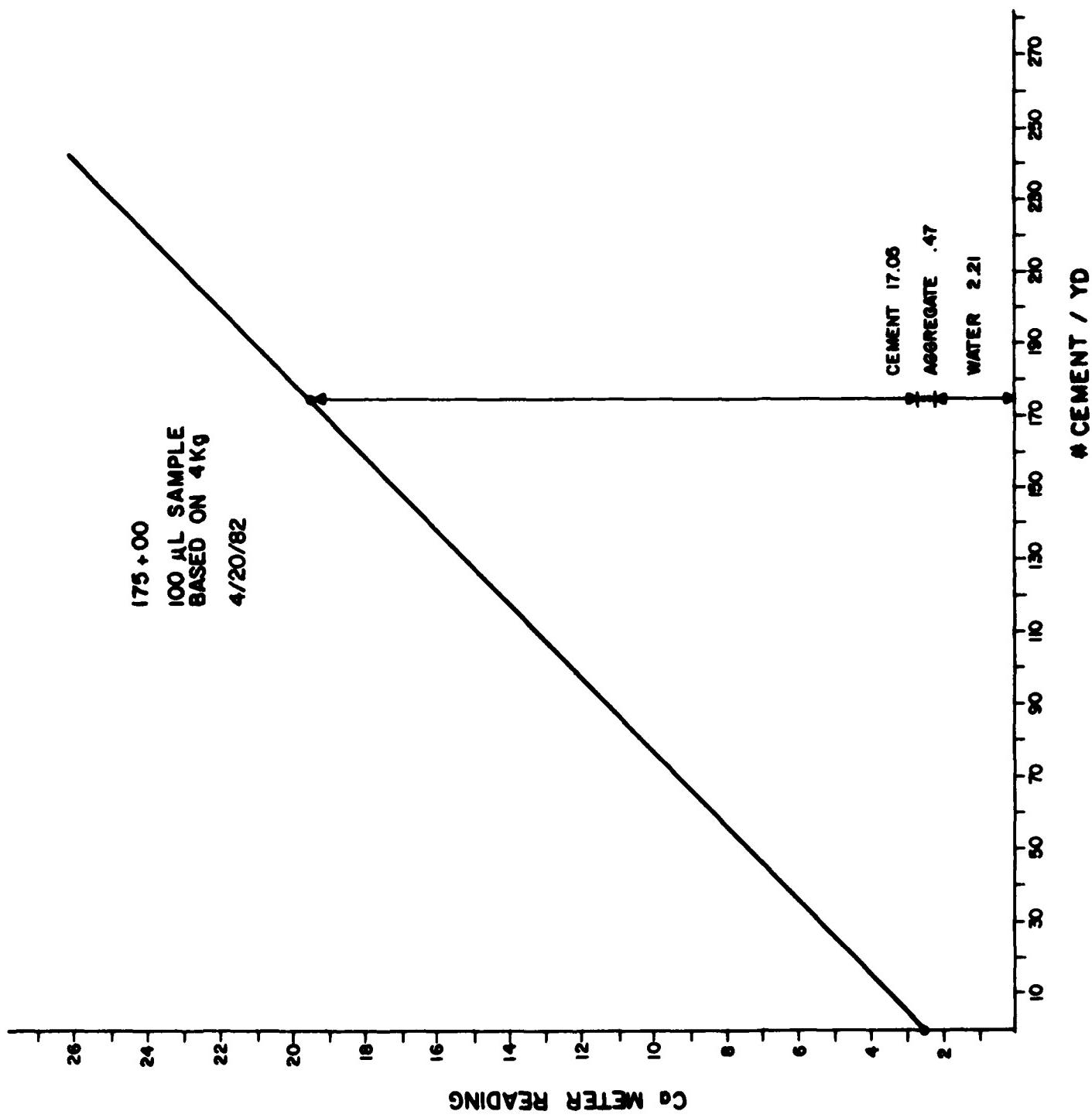
Mix design	% by wt	Ca reading w/H <sub>2</sub> O	Ca reading for raw materials
<b>Mix 80 +32</b>			
3 in.	1245		
1-1/2 in.	843	92.9	2.72 $(2.72 - 2.21) .929 = .47$
3/4 in.	1615		
Sand	163		
Cement	80	1.92 ~ 76.8 g/4 kg	9.98 $(9.98 - 2.21) = 7.77$
Ash	32	.77 ~ 30.8 g/4 kg	2.27 $(2.27 - 2.21) = .06$
Water	182		<u>2.21</u>
Wt.	154.1 pcf		
Total wt	4160	0 cement = 2.74	10.51
<b>Mix 175 + 00</b>			
3 in.	1228		
1-1/2 in.	832	91.4	2.72 $(2.72 - 2.21) .914 = .47$
3/4 in.	1593		
Sand	161		
Cement	175	4.19 ~ 167.7 g/4 kg	19.26 $(19.26 - 2.21) = 17.05$
Ash	—		
Water	185		<u>2.21</u>
Wt.	154.6 pcf		
Total wt	4174	0 cement = 2.68	19.73
<b>Mix 175 + 80</b>			
3 in.	1199		
1-1/2 in.	812	89.4	2.72 $(2.72 - 2.21) .894 = .46$
3/4 in.	1555		
Sand	157		
Cement	175	4.20 ~ 168 g/4 kg	19.29 $(19.29 - 2.21) = 17.08$
Ash	80	1.92 ~ 76.9 g/4 kg	2.35 $(2.35 - 2.21) = .14$
Water	185		<u>2.21</u>
Wt.	154.2 pcf		
Total wt	4163	0 cement = 2.81	19.89
<b>Mix 315 + 135</b>			
3 in.	—		
1-1/2 in.	1634	84.3	2.52 $(2.52 - 2.21) .843 = .261$
3/4 in.	1867		
Sand	—		
Cement	315	7.59 ~ 303.6 g/4 kg	33.15 $(33.15 - 2.21) = 30.94$
Ash	135	3.25 ~ 130 g/4 kg	2.44 $(2.44 - 2.21) = .23$
Water	200		<u>2.21</u>
Wt.	153.7 pcf		
Total wt	4151	0 cement = 2.70	33.64
<b>Mix 330 + 130</b>			
3 in.	—		
1-1/2 in.	—	82.3	3.14 $(3.14 - 2.21) .823 = .765$
3/4 in.	3272		
Sand	—		
Cement	330	8.30 ~ 332 g/4 kg	36.05 $(36.05 - 2.21) = 33.84$
Ash	130	3.27 ~ 130.8 g/4 kg	2.44 $(2.44 - 2.21) = .23$
Water	245		<u>2.21</u>
Wt.	147.3 pcf		
Total wt	3977	0 cement = 3.21	37.045

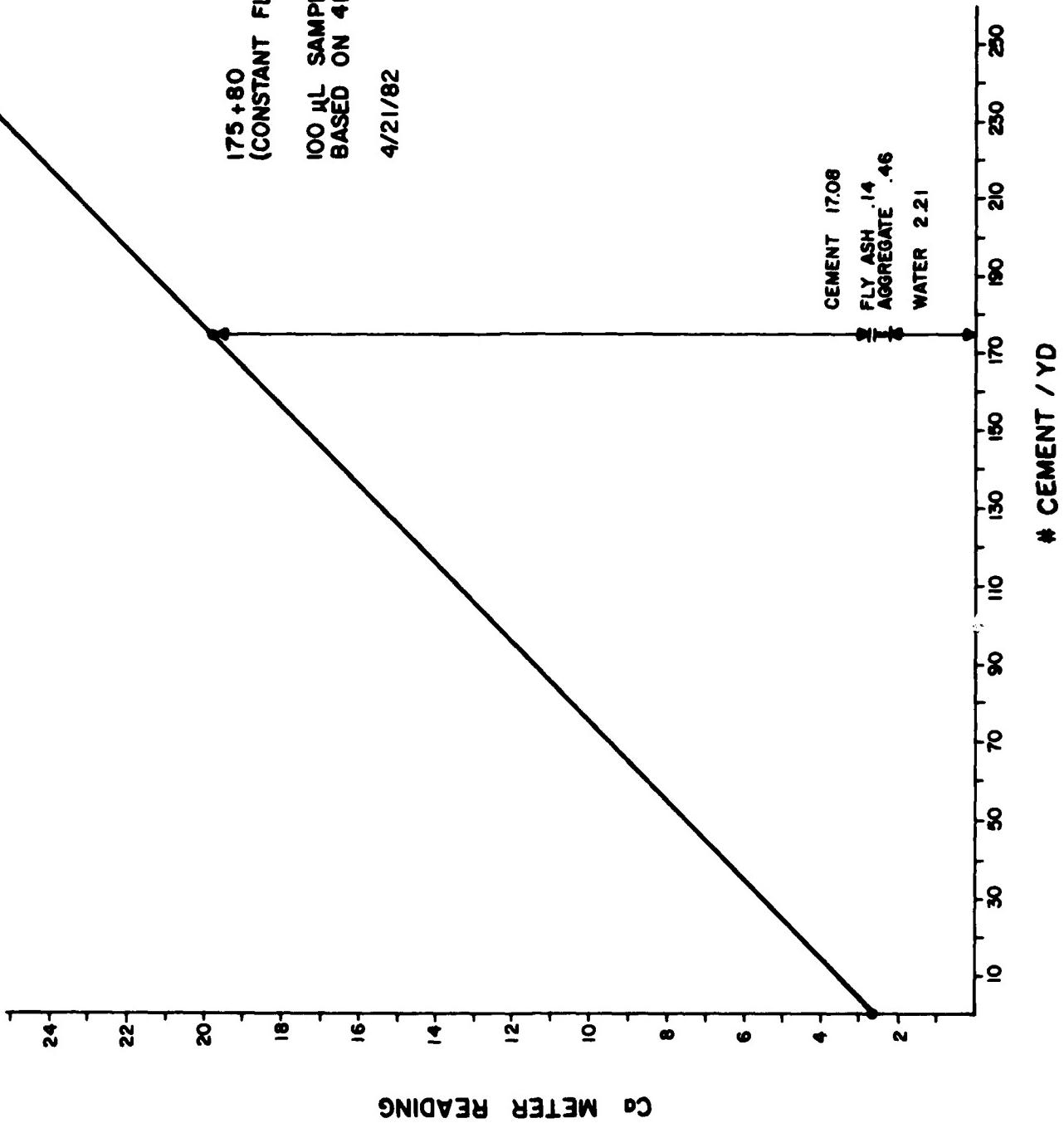
**80+32  
(CONSTANT FLY ASH)**

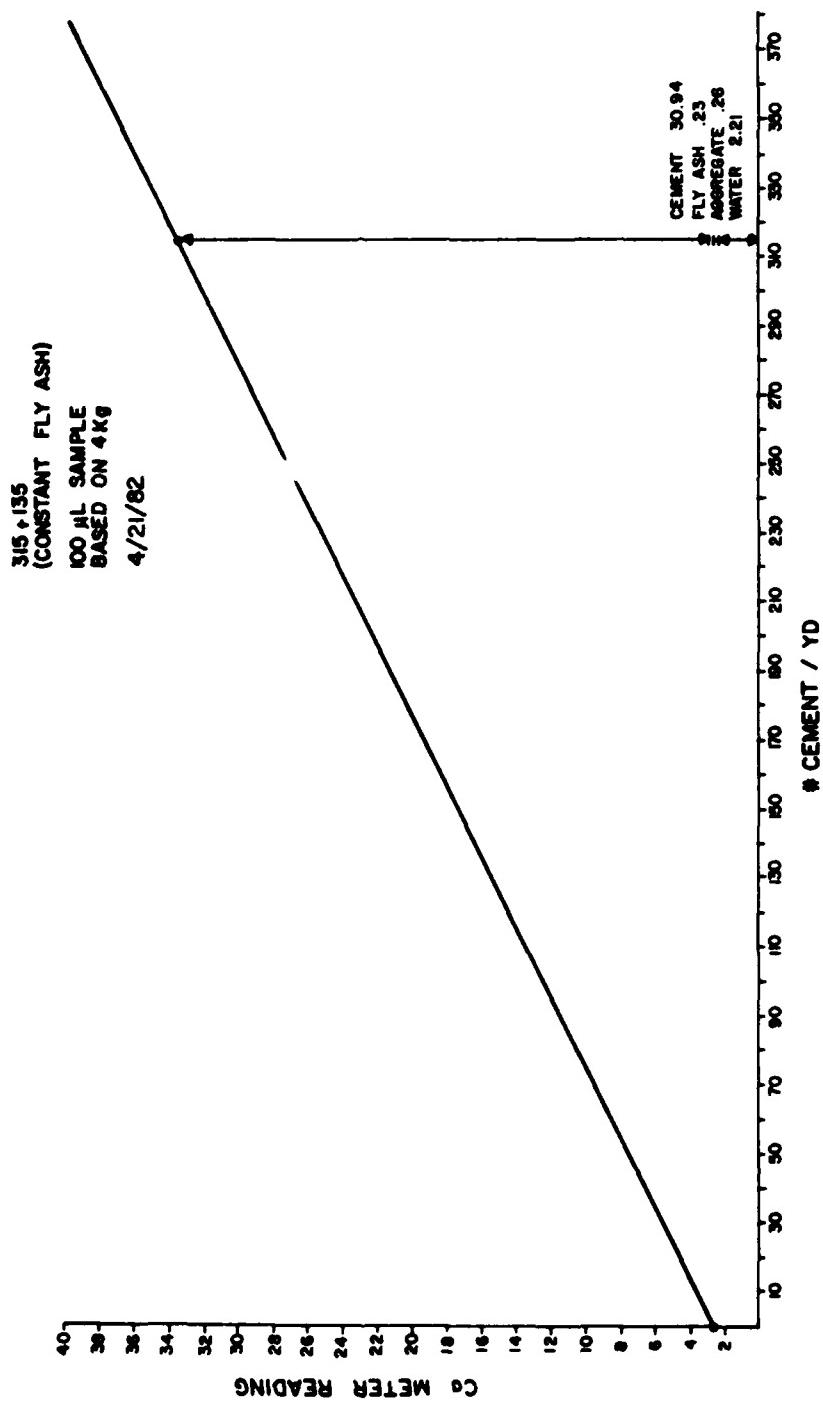
**100 AL SAMPLE  
BASED ON 4KG**

**4/20/82**

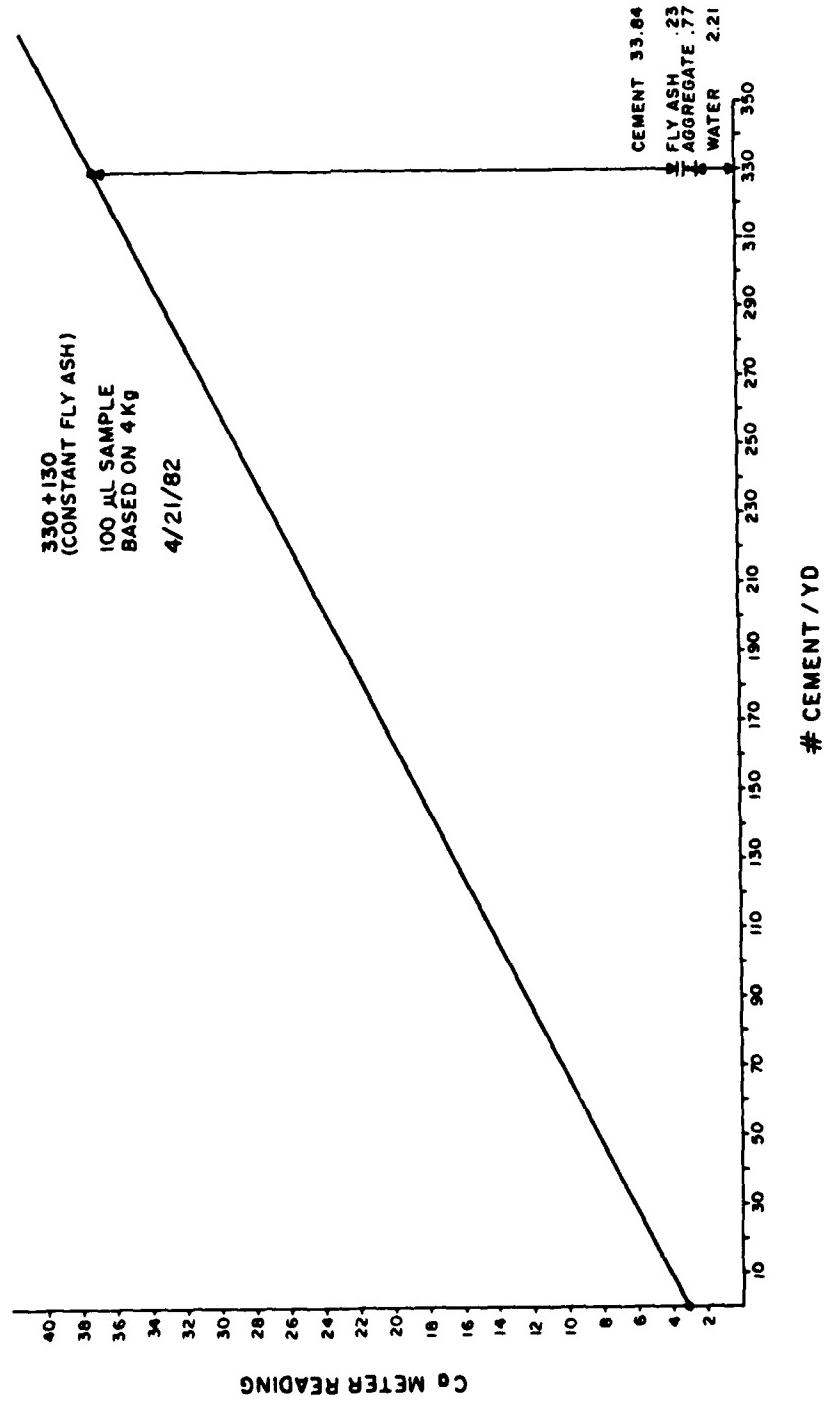








## CALIBRATION CURVES FOR RCC



**APPENDIX E:**  
**SMALL CYLINDERS—DATA SUMMARY**

	CQM cement	CQM ÷ theoretical	CQM cement ÷ cylinder strength			
			3 Day	7 Day	14 Day	28 Day
<b>80 + 32 Mix</b>						
<i>n</i>	37	37	37	37	36	36
$\bar{x}$	77.89	.9736	.2010	.1529	.1029	.0719
SD*	19.12	.2390	.0655	.06633	.0285	.0213
Var	355.56	.0555	.0042	.0043	.0008	.00044
SD/ $\bar{x}$	.2455	.2455	.3259	.4338	.2770	.2962
<b>175 + 00 Mix</b>						
<i>n</i>	45	45	43	43	43	42
$\bar{x}$	172.71	.9869	.2946	.1903	.1347	.1034
SD	34.26	.1958	.1127	.0609	.0399	.0346
Var	1147.9	.0375	.0124	.00362	.00156	.0012
SD/ $\bar{x}$	.1984	.1984	.3826	.3200	.2962	.3346
<b>175 + 80 Mix</b>						
<i>n</i>	48	48	48	47	48	48
$\bar{x}$	178.31	1.019	.2527	.16897	.12493	.09691
SD	32.3	.1847	.0922	.05414	.04312	.04081
Var	1023.3	.0334	.00833	.00287	.00182	.00163
SD/ $\bar{x}$	.1811	.1813	.3649	.3204	.3452	.4211
<b>315 + 135 Mix</b>						
<i>n</i>	40	40	37	38	38	37
$\bar{x}$	296.13	.9401	.2359	.1709	.1307	.1309
SD	43.11	.1368	.0723	.0798	.0568	.0604
Var	1811.8	.0183	.0051	.0062	.0031	.0036
SD/ $\bar{x}$	.1456	.1455	.3065	.4669	.4346	.5813
<b>330 + 130 Mix</b>						
<i>n</i>	12	12	12	12	11	12
$\bar{x}$	302.75	.9174	.4992	.3616	.2222	.1611
SD	30.68	.0930	.1226	.1526	.1133	.0688
Var	862.85	.00792	.0138	.0213	.0117	.0043
SD/ $\bar{x}$	.1013	.1014	.2456	.4220	.5099	.4271

\*SD = Standard deviation.

80 + 32 Mix			175 + 00 Mix			175 + 80 Mix		
Date	CQM cement	CQM ÷ theoretical	Date	CQM cement	CQM ÷ theoretical	Date	CQM cement	CQM ÷ theoretical
5/21	76	.95	6/17	145	.83	6/2	156	.89
6/8	77	.96	6/19	152	.87	6/5	176	1.01
6/14	64	.80	6/21	146	.83	6/10	122	.70
6/16	89	1.11	6/25	184	1.05	6/16	103	.59
6/17	81	1.01	6/28	140	.80	6/19	166	.95
6/21	90	1.12	6/29	192	1.10	6/22	160	.91
6/22	69	.86	7/2	182	1.04	6/25	158	.90
6/24	47	.59	7/9	140	.80		200	1.14
6/26	82	1.02	7/12	160	.91	6/28	173	.99
6/29	75	.94	7/16	175	1.00	7/7	190	1.09
7/3	94	1.17	7/17	160	.91	7/10	200	1.14
7/8	60	.75	7/19	187	1.07	7/14	221	1.26
7/9	67	.84	7/22	218	1.25	7/17	168	.96
7/13	55	.69	7/23	163	.93	7/20	164	.94
7/15	83	1.04	7/24	198	1.13	7/21	204	1.17
7/20	79	.99	7/28	180	1.03	7/23	134	.77
7/22	86	1.07	7/31	180	1.03	7/26	170	.97
7/30	54	.67	8/3	216	1.23	7/28	173	.99
7/31	104	1.30	8/4	196	1.12	7/30	208	1.19
8/3	101	1.26	8/6	219	1.25	8/2	160	.91
8/5	109	1.36	8/10	112	.64	8/3	141	.81
8/7	69	.86	8/11	207	1.18	8/7	160	.91
8/9	89	1.11	8/14	158	.90	8/10	258	1.47
8/12	73	.91	8/16	186	1.06	8/11	136	.78
8/13	105	1.31	8/18	188	1.07	8/13	209	1.19
8/14	73	.91	8/19	208	1.19	8/17	190	1.09
8/18	113	1.41	8/21	114	.65		200	1.14
8/19	88	1.10	8/23	114	.65	8/19	176	1.01
8/26	74	.92	8/25	192	1.10	8/20	215	1.23
8/28	82	1.02	8/27	168	.96	8/24	178	1.02
9/1	80	1.00	8/30	201	1.15	8/26	220	1.26
9/2	58	.72	8/31	174	.99	8/28	200	1.14
9/7	79	.99	9/2	186	1.06	8/30	206	1.18
9/9	38	.48	9/3	160	.91	9/1	146	.83
9/11	35	.44	9/8	96	.55	9/3	158	.90
9/14	70	.87	9/10	142	.81	9/4	188	1.07
9/21	114	1.42	9/13	262	1.50	9/8	188	1.07
			9/15	171	.98		130	.74
<i>n =</i>	35	35	9/16	146	.83	9/10	160	.91
<i>x̄ =</i>	80.3	1.00	9/17	232	1.33	9/14	220	1.26
<i>SD =</i>	16.7	.21	9/18	178	1.02	9/16	186	1.06
<i>Var =</i>	272	.04	9/19	208	1.19	9/17	218	1.25
			9/20	175	1.00	9/18	156	.89
			9/22	130	.74	9/20	130	.74
				131	.75	9/21	210	1.20
						9/23	160	.91
			<i>n =</i>	45	45		242	1.38
			<i>x̄ =</i>	172.7	.99		172	.98
			<i>SD =</i>	34.3	.20			
			<i>Var =</i>	1148	.04	<i>n =</i>	48	48
						<i>x̄ =</i>	178.3	1.019
						<i>SD =</i>	32.3	.18
						<i>Var =</i>	1023	.03

## 315 + 135 Mix

Date	CQM cement	CQM ÷ theoretical
5/24	364	1.16
6/10	278	.88
6/15	260	.83
6/16	310	.98
6/18	261	.83
6/19	350	1.11
6/23	324	1.03
6/24	320	1.02
6/29	312	.99
6/30	302	.96
7/1	252	.80
7/3	360	1.14
7/6	290	.92
7/8	326	1.03
7/10	321	1.02
7/15	308	.98
7/16	318	1.01
7/21	326	1.03
7/27	350	1.11
7/29	350	1.11
8/3	254	.81
8/6	210	.67
8/10	224	.71
8/12	292	.93
8/13	315	1.00
8/14	302	.96
8/20	288	.91
8/21	290	.92
8/23	291	.92
8/25	310	.98
8/27	275	.87
8/31	360	1.14
9/2	264	.84
9/4	353	1.12
9/7	278	.88
9/9	300	.95
9/13	260	.83
9/15	286	.91
9/24	165	.52
	246	.78

$n =$  40      40  
 $\bar{x} =$  296.1      .94  
 $SD =$  43.1      .14  
 $Var =$  1812      .018

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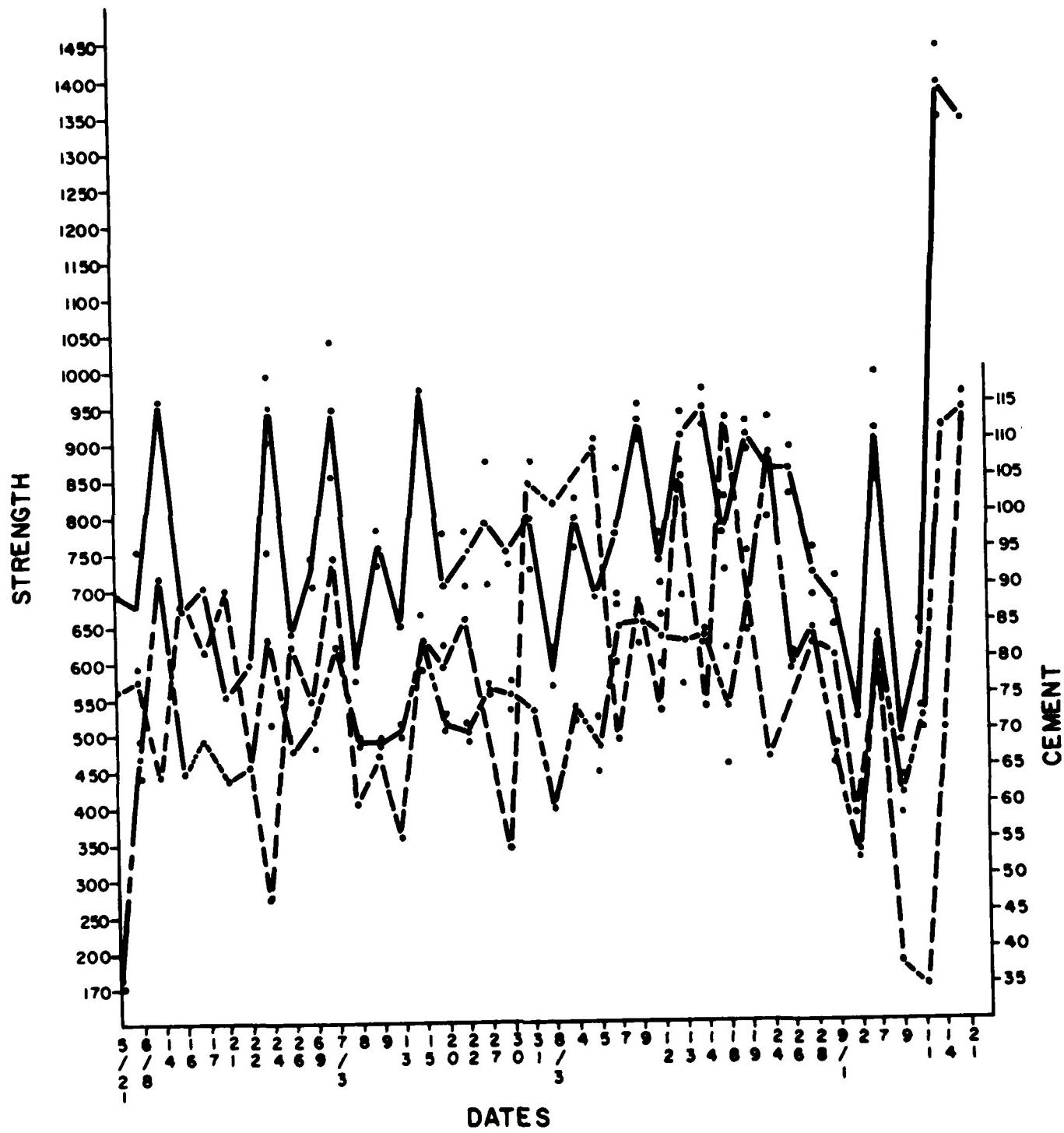
## 330 + 130 Mix

Date	CQM cement	CQM ÷ theoretical
6/14	338	1.02
6/26	308	.93
6/28	330	1.00
7/1	330	1.00
7/6	336	1.02
7/12	300	.91
7/14	299	.91
7/19	270	.82
7/26	252	.76
8/2	322	.98
8/9	294	.89
8/16	254	.77

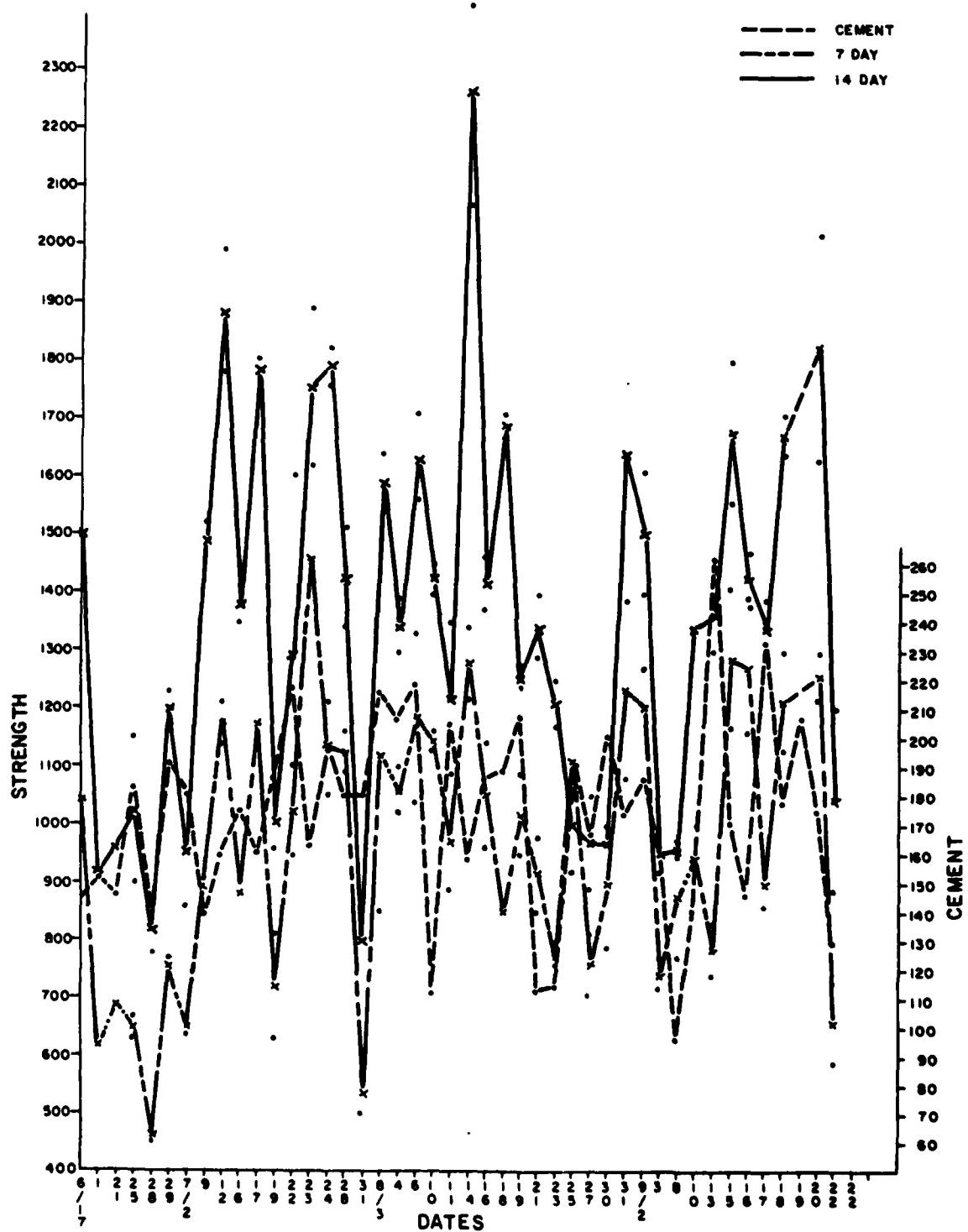
$n =$  12      12  
 $\bar{x} =$  302.8      .92  
 $SD =$  30.7      .093  
 $Var =$  863      .008

**80 + 32 (S)**

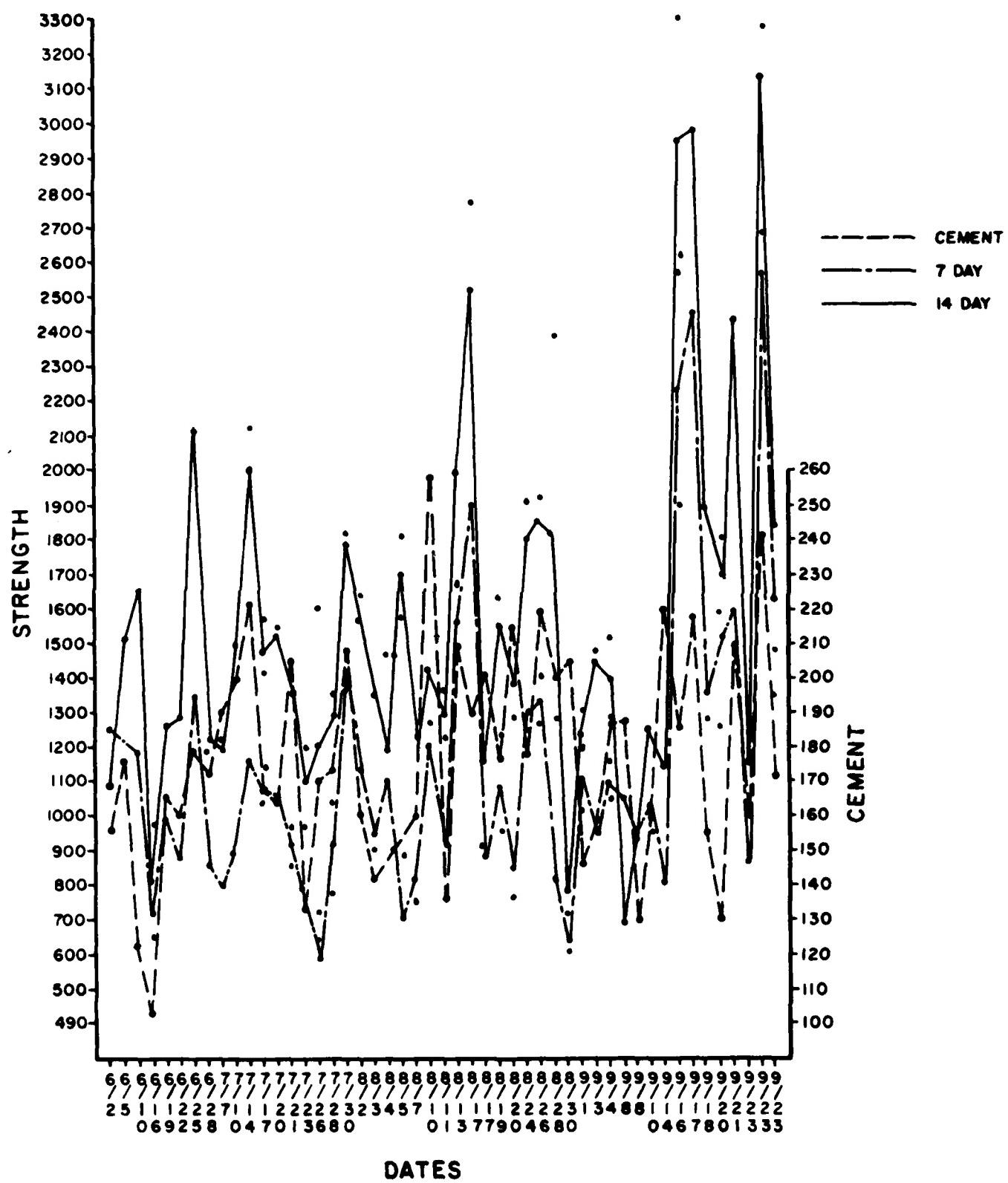
— CEMENT  
— 7 DAY  
— 14 DAY



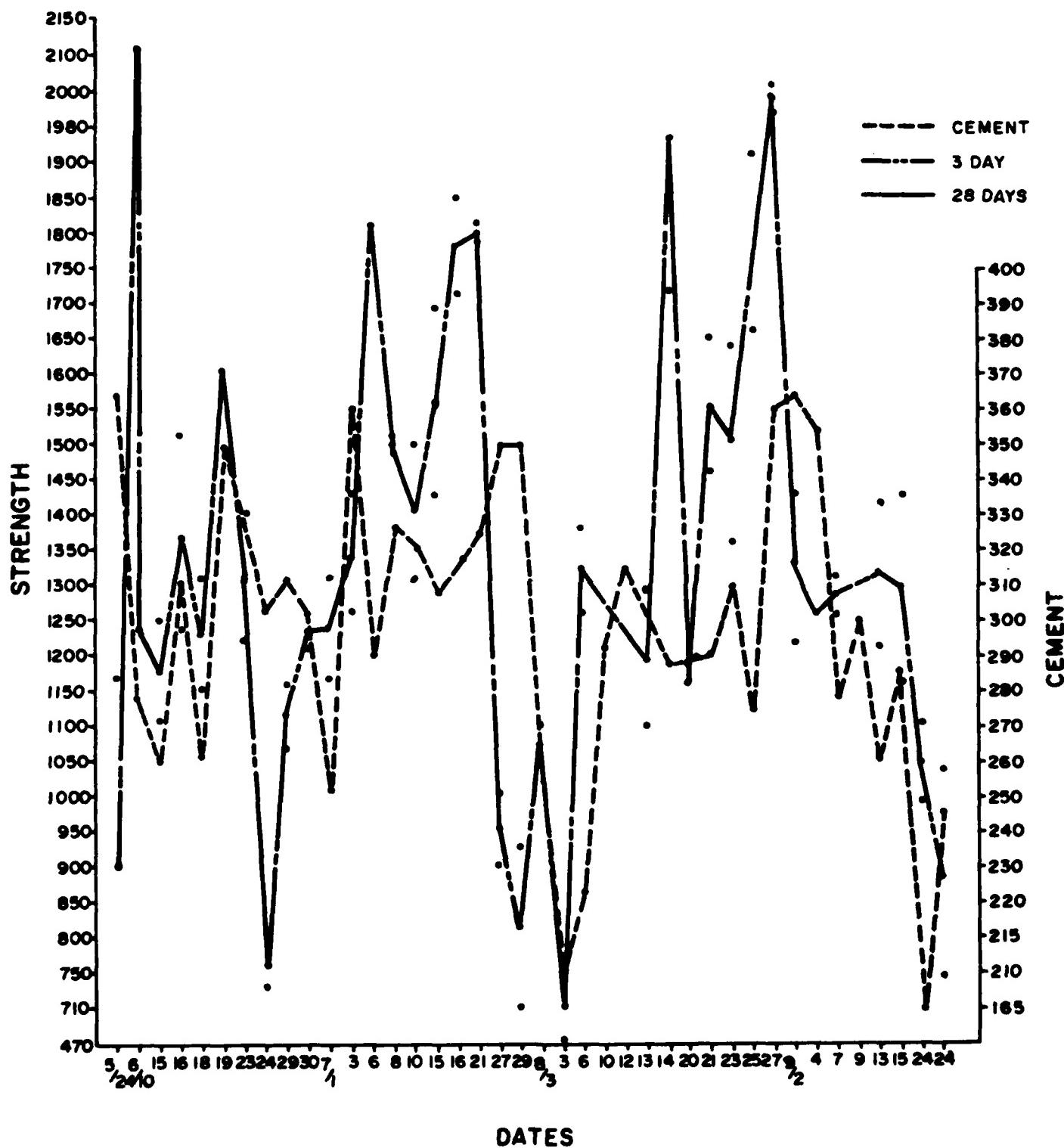
175 + 00 (S)



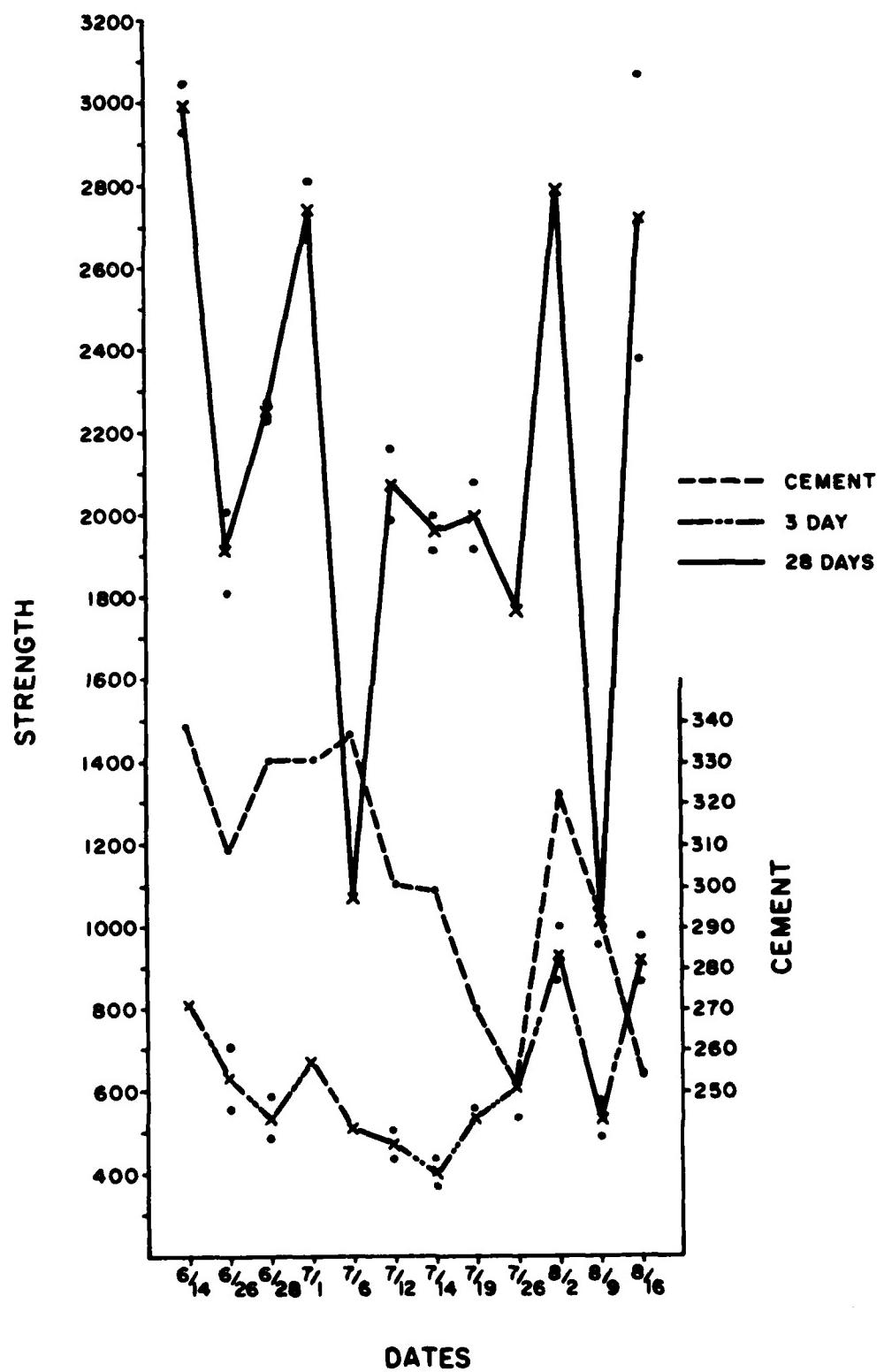
175 + 80 (S)



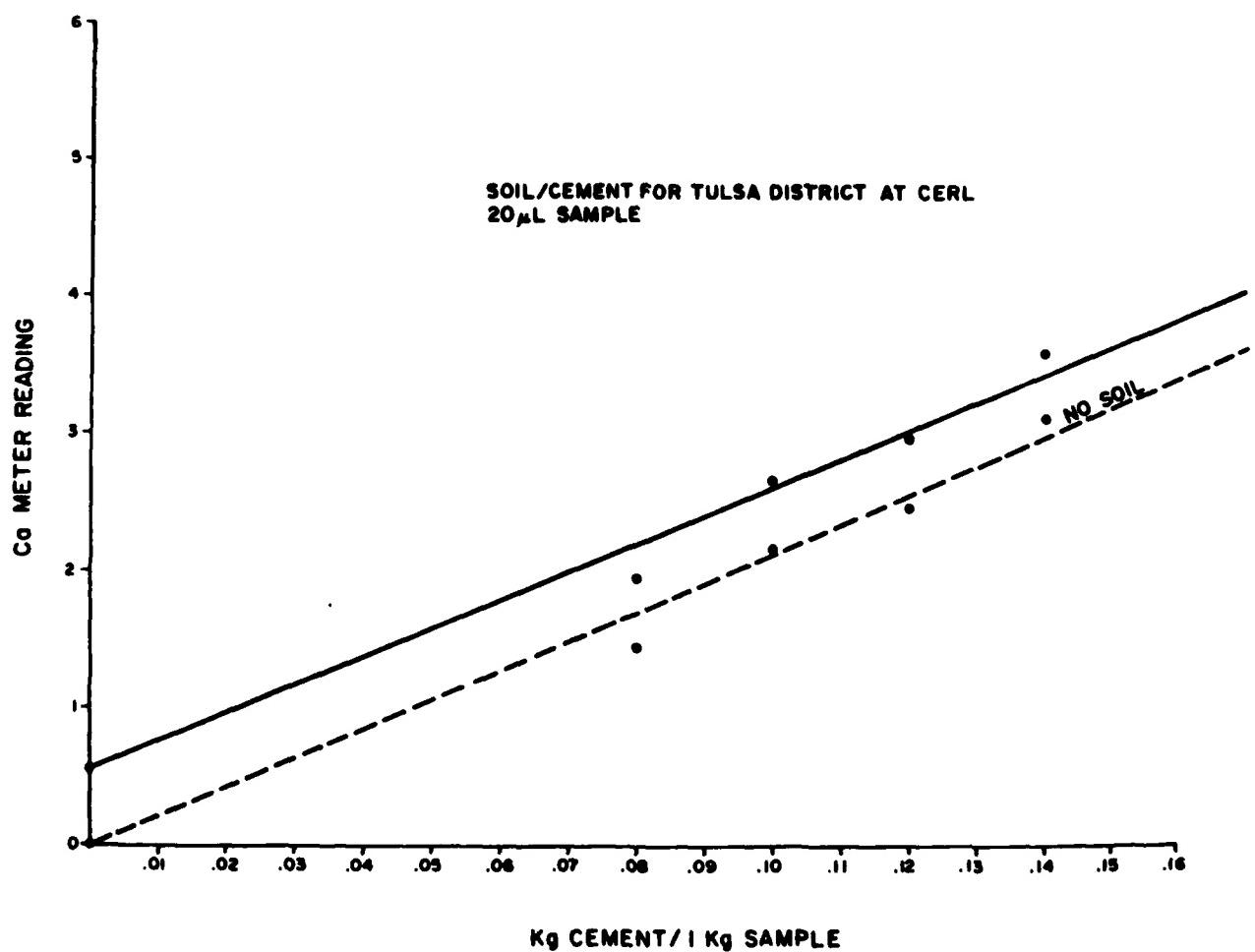
315 + 135 (S)



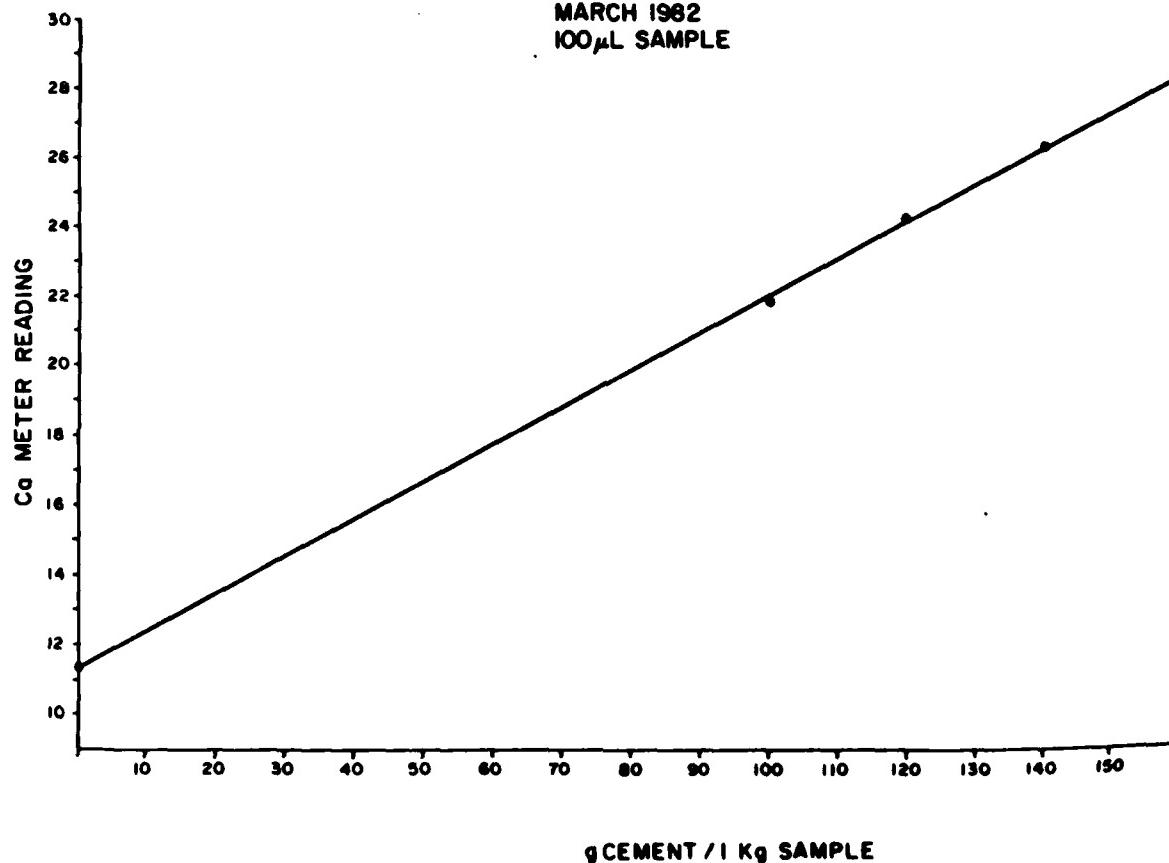
$330 \pm 130(S)$



**APPENDIX F:**  
**CALIBRATION CURVES FOR TRUSCOTT, TX**



SOIL/CEMENT FOR TULSA DISTRICT AT TRUSCOTT  
MARCH 1982  
100 $\mu$ L SAMPLE



**APPENDIX G:**  
**TRUSCOTT SOIL CEMENT DATA**

Truscott report #	Cement content				<u>CQM actual</u>
	Actual %*	CQM %*	Avg		
11	13.18	10.2			.95
		14.0	12.57		
		13.5			
12	12.71	—			—
13	12.90	12.5	13.25	1.02	
		14.0			
14	11.8	—			—
15	11.11	10.5			1.08
		12.8	12.00		
		12.7			
16	12.57	11.7			.89
		10.9	11.23		
		11.1			
17	14.02	12.0			.84
		11.3	11.7		
		11.8			
18	12.82	10.6	10.4		.81
		10.2			
19	12.37	12.2	12.9		1.04
		13.6			
20	12.47	12.3			.99
21	12.28	12.6			1.03
22	12.41	13.4			1.03
		12.4	12.73		
		12.4			
23	11.61	10.8			.93
24	12.57	11.0			.86
		11.4	10.8		
		10.0			
25	12.30	11.3	11.7		.95
		12.1			
26	11.90	10.7	10.7		.90
		10.7			
27	11.85	14.5			1.05
		11.6	12.47		
		11.3			
28	12.04	11.3			.98
		10.9			
		13.3	11.85		
29	12.16	11.9			
		15.3			
		15.5			
		12.1	13.95		

\*Actual cement based on daily scale readings at plant; CQM % based on CQM content determination and estimation of lifts.

Truscott report #	Actual %*	CQM %*	Avg	<u>CQM actual</u>
30	12.13	12.9 13.2 17.3 11.7	14.07	1.16
31	12.03	17.2 10.4 15.8 11.3	13.68	1.14
32	12.32	14.3 12.8 12.0	13.03	1.06
33	12.22	7.9 11.8 10.9	10.2	.84
34	12.18	13.8 10.8 11.9 9.6 12.2	11.66	.96
35	12.24	9.4 12.6	11.0	.90
36	11.91	10.3 11.0	10.65	.89
37	11.72	11.3 9.2 10.5	10.33	.88
38	11.58	11.6		1.00
39	11.34	11.0 11.1	11.05	.97
40	10.98	9.8 10.5 10.4 9.2 12.2	10.42	.95
41	11.46	11.7		1.02
42	11.46	10.0		.87
43	11.07	10.3 9.3 10.6 12.0	10.55	.95
44	11.37	12.6 10.2	11.40	1.00
45	11.00	11.2 11.8	11.5	1.05
46	12.00	—		—
47	11.52	12.7 11.6 11.9 11.4	11.9	1.03
48	11.72	14.0		1.20

Truscott report #	Actual %*	CQM %*	Avg	<u>CQM</u> <u>actual</u>
49	12.22	10.8 12.6	11.7	.96
50	12.12	12.3 12.9	12.6	1.04
51	11.74	9.6 12.1 10.8	10.83	.92
52	11.83	12.0 12.8	12.4	1.05
53	12.06	9.5 10.3 11.3	10.37	.86
54	12.00	13.9		1.16
55	11.69	—		—
56	12.05	10.6		.88
57	11.97	11.6 11.5	11.55	.97
58	11.85	10.8		.91
59	11.8	11.8 11.4 14.6	12.6	1.07
60	11.86	11.8 11.3	11.55	.97
61	11.66	9.8 9.8 11.5	10.37	.89
62	11.93	12.2 10.4	11.3	.95
63	11.74	9.9 12.9 12.5	11.77	1.00
64	11.42	11.5		1.01
65	11.60	13.6 13.0	13.3	1.15
66	11.94	9.6 9.3 10.7 11.0		.85
67	11.69	13.6 11.7	12.65	1.08
68	11.83	11.5		.97
69	11.96	14.5 11.1 11.2	12.27	1.03
70	11.96	10.9		.91
71	11.98	—		—
72	11.81	11.0 14.1	12.55	1.06
73	12.02	13.7 13.3	13.5	1.12

Truscott report #	Actual %*	CQM %*	Avg	<u>CQM</u> <u>actual</u>
74	12.06	—	—	
75	12.00	13.9		1.16
76	11.96	12.7	12.3	1.03
		11.9		
77	12.18	12.0		.99
78	11.9	12.3		1.03
79	11.75	13.8	13.55	1.15
		13.3		
80	11.77	14.7	13.85	1.18
		13.0		
81	11.87	13.7	12.35	1.04
		11.0		
82	11.49	13.1	12.55	1.09
		12.0		
83	11.74	12.1	12.95	1.10
		13.8		
84	11.72	11.3	10.9	.93
		10.5		
85	11.61	—		—
86	11.90	—		—
87	12.29	13.6	12.95	1.05
		12.3		
88	12.04	12.1		1.01
<hr/>				
<i>n</i> =	70			
<i><math>\bar{x}</math></i> =	1.00			
SD =	.094			
Var =	.0088			

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